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NATIONAL BUREAU OF STANDARDS

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1968

Technical Highlights

of the

National Bureau of Standards

Institute for Basic Standards
Institute for Applied Technology

Institute for Materials Research
Center for Radiation Research

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INTRODUCTION

Management Progress

The National Bureau of Standards has continuing responsibility in a number of areas under terms of the Organic Act which created it in 1901 and through subsequent amendments to the Act. In addition, as a result of changing technology and through special legislative acts, new and expanding activities have been undertaken over the years. Changes in Bureau activity have occasionally resulted in redirection of administrative and management emphasis; the most recent major reorganization came in 1964 with the formation of four Institutes to encompass the various activities that existed at that time.

The significant changes that have taken place in the past fiscal year are discussed below. In each case, the change was designed to make the Bureau more responsive to needs of the commercial, scientific, and industrial groups which it serves.

Center for Radiation Research Created

A Center for Radiation Research was formed to provide more effective management of the Bureau's large radiation facilities, previously under two different Institutes in the NBS structure. With all of its major radiation-producing machines under central management the Bureau will be able (1) to provide special services to all of its programs, (2) to carry out more vigorous basic research and (3) to share its unique resources with other Government agencies and with universities.

Special radiation facilities at NBS include a ten megawatt nuclear research reactor and a 100 million electron volt linear accelerator, as well as a number of other radiation-producing machines and sources. These facilities, especially the large machines, are used in a wide variety of studies by various units in the Bureau, and are available to the Government and academic communities of the Washington area. NBS studies cover radiation measurement and standards, basic nature of matter and radiation, and the effects of radiation on matter and materials.

The new Center for Radiation Research is composed of a Reactor Radiation Division, Linac Radiation Division, Nuclear Radiation Division, and Applied Radiation Division.

Reorganization of Boulder Laboratories

During the fiscal year the NBS Boulder, Colo., Laboratories were unified under a single, local management for the first time since 1964, when the Bureau's programs were divided up between the Institute for Basic Standards (IBS), the Institute for Materials Research (IMR), the Institute for Applied Technology (IAT), and the Central Radio Propagation Laboratory (CRPL). The last has since become part of the Environmental Science Services Administration.

A reorganization which began in midyear was completed June 30, bringing all of the Boulder elements into the NBS Institute for Basic Standards, organized to provide "the central national basis for a complete, consistent system of physical measurement, coordinated with those of other nations."

Transfer of the Boulder Cryogenics Division into IBS and the appointment of a Deputy Director for IBS-Boulder to direct the activities of five research divisions and the services of three support divisions, created a common bond in fulfilling the IBS assignment to develop and maintain standards for physical quantities and for the measurement of physical properties.

New Institute Directors Named

New Directors for the three NBS Institutes were named during the year. The average age of the new Directors is 44, indicative of management policy to fill senior positions with young persons who are scientifically well qualified but also attuned to the expanding and changing role of science and technology in the Nation.

Dr. Ernest Ambler, Chief of the NBS Inorganic Materials Division was named to head the Institute for Basic Standards. Dr. Ambler, a Briton by birth, came to the United States in 1953, and joined NBS the same year. He began performing magnetic research and nuclear orientation studies at temperatures approaching absolute zero and, within eight months after coming to the Bureau, completed the first nuclear alinement experiments to be conducted in the United States.

Dr. Ambler received his B.A., M.A., and Ph. D. degrees at Oxford University in 1945, 1949 and 1953. He shared in the 1964 Samuel Wesley Stratton Award for work demonstrating that the quantum mechanical law of parity conservation does not hold in weak interactions. He was also honored for this work with a Department of Commerce Gold Medal Award in 1957, the Washington Academy of Sciences Award in 1957, and the John Price Wetherill Medal of the Franklin Institute in 1962. In 1960, Dr. Ambler was named one of the ten outstanding young men in the Federal Government, receiving the Arthur S. Fleming Award in recognition of his outstanding work in science.

Dr. John D. Hoffman heads the Institute for Materials Research. He has been with NBS since 1954, when he joined the Polymer Structure Section as a physical chemist. In 1956, a Dielectrics Section under Dr. Hoffman's direction was established to investigate such areas as dielectric properties of polymers, precision dielectric measurements, and theoretical studies on relaxation phenomena in molecular crystals. In 1964 he was named Polymers Division Chief. Dr. Hoffman received his B.S. in chemistry at Franklin and Marshall College in 1942; and in 1948 and 1949, he received his M.S. and Ph. D. degrees in physical chemistry from Princeton University. Dr. Hoffman was presented the 1967 Samuel Wesley Stratton Award. He was corecipient, in 1961 of an NBS Special Act of Service Award. The Washington Academy of Sciences awarded him a certificate of recognition in 1962 "for distinguished service in the science of polymer physics."

Dr. Lawrence M. Kushner was named Director of the Institute for Applied Technology. Before becoming Deputy Director of the Institute in July 1966, Dr. Kushner was Chief of the NBS Metallurgy Division. He joined the Bureau in 1948 as a physical chemist in the Surface Chemistry Section, later becoming Assistant Chief of the Section. In September, 1956, he was appointed Chief of the Metal Physics Section. Dr. Kushner's main scientific research interests are in the relations of defects in the crystal structure of metals to the metal's physical properties.

As a Commerce Science and Technology Fellow, Dr. Kushner worked in the office of the Assistant Secretary of Commerce as a Special Assistant for Legislation in 1964-65. Dr. Kushner was graduated from Queens College with a B.S. in chemistry in 1947. He received both his A.M. and Ph. D. in physical chemistry from Princeton University.

Special Programs

Research Associate Program

The Research Associate Program of the National Bureau of Standards makes it possible for qualified scientists and engineers from industrial corporations, technical trade associations, professional societies, and other Government agencies to work on a full-time basis for finite periods of six months to two years or more at NBS. They work on research programs of mutual interest to the sponsoring group and the National Bureau of Standards. Their salary continues to be paid by their parent organizations.

The Research Associate Program has been in operation for over fifty years but has received renewed emphasis since 1964. Primary responsibility for the development of the program was given to the NBS Office of Industrial Services late in that year. In the past, the

program was primarily aimed at serving the needs of researchers from technical associations. The program is now being aimed directly and primarily at industrial corporations performing research and development. The acceptance by industry of this opportunity, as reflected by success to date is most encouraging.

As of June 30, 1968, there were 61 Research Associates at NBS, 29 of whom came in during fiscal 1968. The total is approximately twice as many as there were in November 1963. Further expansion of the program is planned through the efforts of the NBS Office of Industrial Services with participation by management and technical staff from the three NBS Institutes. The ultimate goal is to have about one Research Associate for every ten NBS professional staff members, or about 150 Research Associates.

An all-day meeting was held between representatives of NBS and about 60 manufacturing concerns in the Pittsburgh area. The purpose was to introduce the Bureau and its services to a cross section of industry in the area. The reactions to this meeting indicate that the approach can be a most fruitful one to use in many cities of the U.S.

Foreign Scientist Visitation Program

For many years, the National Bureau of Standards has been engaged in international activities in various ways. It is in the interest of this country to foster sound systems of standardization and measurement in other countries and for this reason NBS cooperates with international laboratories and organizations engaged in pursuits similar to its own. It is in this spirit that the Bureau receives qualified workers from abroad or that NBS staff are sent to other countries.

A few examples illustrate the general principles listed above. NBS staff are active in certain international organizations such as the International Bureau of Weights and Measures, the International Standards Organization and similar groups which create the definitions upon which most international systems, measurement, and standardization are based. These organizations are found in both developed and less developed countries.

In the less developed countries, the role of NBS is usually to help establish advanced systems in laboratories. NBS has been often called upon to send experts or teams to various parts of the world where need is felt for a standards laboratory or for a sound system of weights and measures. Examples in the recent past are: Iran, South Korea, Saudi Arabia, and Taiwan.

The Bureau recently participated in a cultural exchange with the USSR on standards and norms. For this purpose a staff member of NBS served as a member of the delegation going to the Soviet Union and NBS received the Soviet delegation in December 1967.

As part of the international program, the Bureau in the last year received 732 foreign visitors, 24 foreign guest workers from 16 countries, and six foreign trainees for periods ranging from 2 weeks to 6 months. Included among these guest workers are fellows from United Nations, North Atlantic Treaty Organization, International Atomic Energy Agency, and other national laboratories. NBS staff members participated in a great number of international conferences, at many of which they were asked to deliver papers or chair sessions.

The operations described here constitute an effective way of transferring technology to less developed countries. With the present importance of such transfer the relevance of all international exchanges (documents, persons, instruments, or standard samples) cannot be sufficiently emphasized.

Utilization of Federal Laboratories

During the 2nd session of the 90th Congress, the Subcommittee on Science, Research, and Development of the House Committee on Science and Astronautics held a series of hearings on the Utilization of Federal Laboratories. The purpose of the Subcommittee hearings, chaired by Congressman Emilio Q. Daddario, was to determine "how we can make the best use of our existing Federal laboratories." NBS Director Allen V. Astin was invited to testify and did so on March 27, 1967. Excerpts of Dr. Astin's comments before the Subcommittee appear below.

While the basic measurement mission has provided the prime focus of NBS activities through the years, "the Bureau also has served since its inception as a central resource of scientific and technical competence within Government. Other agencies have made extensive use of the Bureau's competence and facilities, particularly in the exploratory stages of new technical endeavors. This has been a constantly changing role that has involved the Bureau, at least temporarily, in a great variety of technical activities, some of which have continued and have grown to become major national programs involving new Federal laboratories. . . ."

"We do a considerable amount of work for other Federal agencies, ranging from fairly fundamental research to testing and calibration. A substantial part of the NBS annual budget—currently 40 percent of the total—is made up of funds transferred to us from other agencies for services rendered."

"NBS laboratories also serve State and local needs, primarily in the weights and measures field."

"NBS interactions with the academic community are varied and growing. They include use of our laboratory facilities in a number of ways."

“NBS works very closely with private standards making bodies, of which there are some 500 in the United States.”

“In 1961, NBS sponsored the establishment of the National Conference of Standards Laboratories, and has acted as its secretariat ever since. The NCSL is a continuing, nonprofit, laboratory-oriented organization to promote cooperative efforts toward solving common standards and measurement problems. Its members include standards laboratories of individual companies, universities, independent laboratories, and those in other Government agencies.”

“Finally, our laboratory facilities are shared with industry itself. The most effective mechanism is our Research Associate Program,” see page 3.

Legislative Report

Four proposed legislative acts affecting the Bureau were considered by the Congress during the fiscal year. The Flammable Fabrics Act (PL 90-189) was amended on December 14, 1967. The Fire Research and Safety Act of 1968 became law on March 1, 1968. The Standard Reference Data Act was signed into law on July 11, 1968. Also, a bill authorizing the Secretary of Commerce to conduct a study of the metric system was under consideration. (This law was subsequently passed by the Congress and signed into law August 9, 1968.)

Flammable Fabrics Act

The 1967 amendments to the Flammable Fabrics Act (PL 90-189) make certain provisions for a more effective attack on the overall problem of deaths, injuries, and economic losses resulting from fires involving fabrics and related materials, and products made from them. They extended the coverage of the Act to include all articles of wearing apparel and, if the need is shown, items of interior furnishings. They provide the authority for establishing standards and required levels of performance, with certain operational safeguards; the conduct of investigations of the extent of deaths, injuries and economic losses resulting from fire involving garments and interior furnishings; research into the flammability of fabrics and related materials; the development of test methods and revised or new standards; training in the use of the test methods and devices; and provisions for the enforcement of the amended Act.

Under the Act, the Department of Commerce, mainly through NBS, is responsible for research into the flammability of materials and the feasibility of reducing that flammability, development of test methods and apparatus, training in their use, and for the establishment of standards and regulations concerning fabrics and related materials as used in wearing apparel or interior furnishings. The legislation requires the Secretary of Commerce to consider the results of research

and investigations in determining that a hazard exists and that a standard or regulation, or a revision thereof, is needed to protect the public.

Fire Research and Safety Act

The Fire Research and Safety Act of 1968 (PL 90-250) authorizes the Secretary of Commerce to conduct, or through contracts or grants to carry out:

1. Investigations of fire causes, severity, frequency, or other pertinent factors;

2. Research into the causes and nature of fires and the development of improved methods and techniques for fire prevention, fire control, and reduction of death, personal injury, and property damage;

3. Educational programs to inform the public of fire hazards and fire safety techniques and encourage the avoidance of such hazards;

4. Fire information reference services including the collection, analysis, and dissemination of data and research results related to fire protection, control, and reduction of deaths or property loss.

5. Educational and training programs for among other things the improved efficiency, operation, and organization of fire services; and

6. Demonstration projects in support of fire prevention, fire safety principles in constructions, and improved fire services.

Further, the Secretary is to support the development of fire safety and protection engineering or science curriculums, and fire safety courses, seminars, or other instruction materials and aids for courses of instruction. NBS will play a significant role in carrying out these activities.

Standard Reference Data Act

A National system for providing badly needed scientific data to science and industry was authorized by the Standard Reference Data Act which became law on July 11, 1968.

The law authorizes NBS to coordinate the program. The Bureau has had a standard reference data operation for three years, but additional authority given by the new law will strengthen and increase the effectiveness of the system. The main purpose of the standard reference data program is to make readily available to the Nation's scientists and engineers data on the properties of matter and materials—melting points, electrical resistance, strength, and many more. Such data are essential for picking the proper material for making a space probe or a toaster, or any other manufactured item.

TWO KEY STANDARDS PROGRAMS

The role of the National Bureau of Standards as a source of scientific and technical expertise to industry, commerce, science, and Government is clearly evident in two key programs designed to provide highly reliable information and materials to users throughout the world.

A STANDARD REFERENCE DATA PROGRAM provides critically evaluated quantitative information relating to a property of a definable substance or system.

A STANDARD REFERENCE MATERIALS PROGRAM provides well-characterized materials that can be used to calibrate a measurement system or to produce scientific data that can be readily referred to a common base.

These two programs have much in common, as their titles imply. Both are the result of ready-made measurement. Both produce a highly reliable product which foster a user's capability to make meaningful measurements. Both are economically efficient—reference data because they are not exhausted by continued use and reference materials because they are accurately reproducible on a mass production basis. And most important, both serve a critical and wide-spread measurement need in the United States and are important in the development of new areas of international standardization.

THE NATIONAL STANDARD REFERENCE DATA SYSTEM

Improvement in the effectiveness of the Nation's system for scientific and technical information is currently a matter of great popular concern. Much is being said and written about the flow of information from the generator to the user, and much is being done to try to speed the process.

The Chemical Abstracts Service of the American Chemical Society is in the midst of a long-range program designed to increase the retrievability of information within its concern [1]. The American Institute of Physics has embarked on a comprehensive study of means to make the world's output of information in physics more readily available to individual users [2]; the Engineers Joint Council has

a similar program [3]. The Atomic Energy Commission, the Department of Defense, the National Aeronautics and Space Administration, and other major Federal agencies are all increasing their efforts to improve the use of information generated within their technical programs. The President's Office of Science and Technology and the President's Science Advisory Committee maintain continuing efforts to review developments and to stimulate new initiatives for improvement. Indeed, a report of the President's Science Advisory Committee has provided basic guidelines for the development of governmental activities since its appearance five years ago [4].

History of the Program

Government interest in all these activities is coordinated through the Federal Council for Science and Technology by means of its Committee on Scientific and Technical Information (COSATI). The initiative of COSATI and its parent council led to establishment in 1963 of the National Standard Reference Data System, a federal inter-agency activity concerned with one aspect of the broad problem of scientific and technical information—that of providing better access by the American technical community to compilations of critically evaluated data on the properties of substances.

Such compilations have been among the basic tools of scientists and engineers throughout the history of technology; each practitioner owns at least one handbook containing table after table of data on the properties of the substances and systems that he deals with daily. Systematic compilations of data also contribute in a fundamental way to progress at the forefront of science. Samuel Goudsmit [5] recently emphasized this importance with the following words:

“Experimental results in measurements are the backbone of physics. No theory is acceptable unless it agrees with the experimental data. Conversely, a systematic study of experimental results can suggest new theoretical approaches. Tables and graphs of numerical data therefore play an important role in the progress of physics. . . . It is thus obvious that specialized data compilations are of great importance and should have the full cooperation of those producing the data. It is also clear that modern computer techniques can handle such data more efficiently than old tabulations could, especially since their number and variety are growing so rapidly.”

Since the numerical data that result from measurements of properties normally appear somewhere in the world's literature, why not let the individual scientist or engineer look them up whenever he needs a value? There are two major reasons why this procedure is not efficient. First, it is often very difficult to locate a desired value among the millions of papers stored in a technical library; searching indexes, abstracts, and papers can consume many hours. Second, conflicting values

for the same property are often reported. Unless the user is a specialist in the field, he will have difficulty deciding which value he should use. These inefficiencies translate directly into wasted effort and dollars. If the average scientist or engineer were to save only ten minutes a week that he now spends finding and evaluating data, the annual saving to the Nation's research and development activity would be of the order of \$100 million. This estimate takes no account of the benefits of having better data, evaluated by an expert in the field, at hand when needed. Obviously, very significant economic benefits can be readily obtained through a coordinated, comprehensive program for reviewing the literature, extracting and evaluating the property data contained therein, and disseminating them in convenient form.

Because of their usefulness and economic benefits, many compilations of data [6] have already been produced throughout the world. However, existing mechanisms have not been able to keep pace with the flood of new data appearing in the literature, except in a few specialized areas. Some compilations are "one shot" projects, resulting in products that are never updated, others have been continuing activities lasting many years. Some have been sponsored by mission-oriented agencies of the United States Government; others, by private organizations. However, many newly recognized properties are not covered at all, and the time lag between the appearance of data in original literature and their evaluation for inclusion in a critical compilation has been rapidly increasing. Even in the areas covered by active projects there has been little coordination or standardization of format and quality, and in some technical areas there is extensive duplication.

Recognizing the deficiencies of the existing situation and the stake of the U.S. Government in the financial support of the Nation's research and development activity, COSATI decided that a government-wide coordinated effort was needed; thereupon it recommended that a proposed plan of action for increasing the level of effort of the National Bureau of Standards in this field be expanded to encompass the total Federal effort within all agencies, with administrative responsibility assigned to NBS. Adopting this recommendation, the Federal Council for Science and Technology and the President's Office of Science and Technology promulgated a Federal policy establishing the National Standard Reference Data System (NSRDS).

The NSRDS is regarded as a subsystem within the concept of the national measurement system [7], which is envisioned as comprising a central core of national standards of measurement, a set of consistent instruments (calibrated through appropriate application of the national standards), a body of reference data that provides users with readymade answers to questions on the properties of substances,

and finally the entire set of meaningful measurements made throughout science, technology, and the economy. From this viewpoint, NSRDS is a portion of the activities leading to dissemination of readymade data for use by the technical community of the United States.

Responsibilities of NBS

In accepting the charge from the Federal Council for Science and Technology, the Bureau has taken responsibility for (i) increasing the availability of reliable reference data by sponsoring critical evaluation and data compilation projects as needed, (ii) coordinating related work under the auspices of all Government agencies, (iii) establishing standards of quality for products of the system, (iv) operating a national center for standard reference data, and (v) establishing standards of methodology and such other functions as are required to ensure the compatibility of all operational components of NSRDS. The goals of NSRDS are to be achieved through operation of an integrated network of data-evaluation centers and related projects located wherever special technical competence for a particular project may exist.

Since data can be adequately evaluated only by specialists whose judgments are accepted by their peers, each data center is to be concerned with a carefully delimited technical scope; normally it will be established as an adjunct to the work of an individual or group having an established reputation for competence and vigor.

Guidelines have been established to limit the boundaries of NSRDS and restrict its program to a manageable size. The program is to be concerned with (i) the data of physical science only (data relating to biological phenomena will be excluded); (ii) well-defined substances only (substances whose composition, structure, and energy content are well enough known that measurements of the property under consideration do not wander erratically); and (iii) well-defined properties that are intrinsic to the substance or system being studied (properties that must be defined in terms of the system used for measurement—such as Brinell hardness and Charpy breaking strength—are excluded).

Operation of the System

Within the National Bureau of Standards the responsibility for administering NSRDS has been assigned to the Office of Standard Reference Data, created for that purpose within the Institute for Basic Standards. Three major groups of activities within the Office of Standard Reference Data have been initiated; these are concerned with (i) the planning and implementation of projects for compiling

data, organized into several broad technical areas; (ii) an information-systems design and research activity; and (iii) various specialized information services to be provided to the technical community.

For program management the data-compilation projects of the Office of Standard Reference Data have been subdivided into seven broad subprograms: (i) nuclear properties, (ii) atomic and molecular properties, (iii) solid-state properties, (iv) thermodynamic and transport properties, (v) chemical kinetics, (vi) colloid and surface properties, and (vii) mechanical properties. In each, responsibility for developing a comprehensive, coordinated program has been assigned to a program manager.

Existing projects of other governmental and nongovernmental agencies are taken into account, and project priorities are determined by consultation with groups of specialists from the academic world, Government, and industry. Some of the projects are conducted within the experimental divisions of the Bureau, others in university laboratories or in other Government laboratories, and a few, by industry. None is under the direct operational supervision of the Office of Standard Reference Data, which is intended to provide program management exclusively.

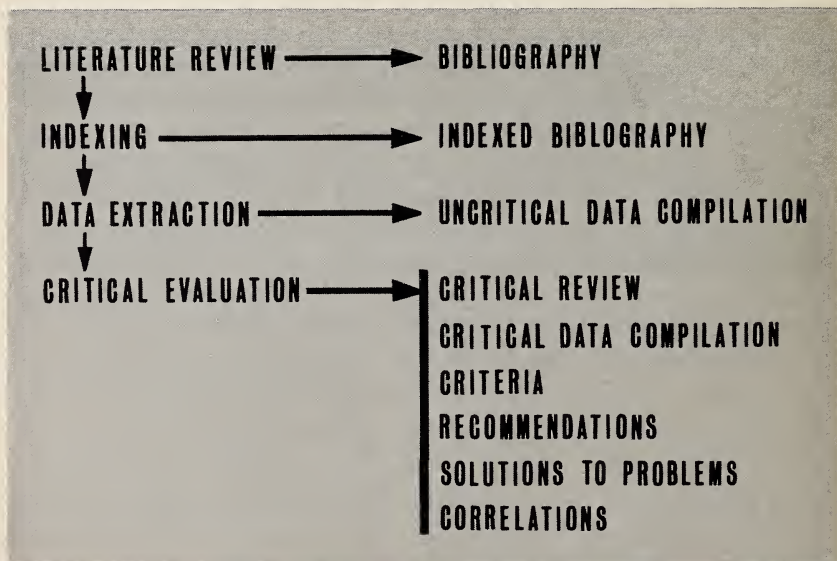
The raw material for any data compilation project is the results of measurements by scientists and engineers throughout the world. Normally, these results are reported in the literature, but some will appear in journals which may be obscure or difficult to obtain. Moreover, an increasing fraction of results worth saving now appears in Government reports. In some areas (data on neutron cross sections are one example) many of the data generated in the laboratory never appear in any report or publication. In such instances the compiler personally may have to pry the data from the measurer. For a specific case, the degree of comprehensiveness that can be achieved must be a practical compromise between the desired 100 percent and the cost in time, money, and effort of achieving that goal. For most existing projects the comprehensiveness probably reaches 90 to 99.8 percent.

The procedure for "critical evaluation" varies widely from project to project. In present practice in some data centers, the experimental technique is reviewed, calculations are spot checked, values of the fundamental constants are checked to ensure that the latest values were used, the temperature scale is checked (if appropriate) and limits of experimental uncertainty are independently assessed. In other centers, the data evaluator may decide, for intangible reasons that he may find difficult to formulate, that one particular value in the literature is "better" than another value. Such a judgment by a specialist of broad experience should not be underrated; the value obtained is much more likely to be accurate than the result of unweighted averaging. Most people agree that the first procedure provides a better "critical evalua-

tion" than the second. However, for the practical purposes to which many compilations are applied, such a review is not justified, and the second procedure, or an intermediate one, is employed.

The question arises then, what degree of critical evaluation is required for a compilation to be considered "standard" reference data? It is probably desirable to use the word "standard" sparingly; it has connotations that apply to few existing compilations. For the present, when measurement results for most properties are uncertain and many are in dispute, the shorter term "reference data" would avoid the implications aroused by use of the word standard. "Standard reference data," the ultimate goal of NSRDS, are to be striven for constantly, but perhaps not reached in many fields for years. Because of the variation in procedures for critical evaluation, all publications of NSRDS are to describe the criteria used for judgment and the argumentation used to derive the recommended values.

For each individual compilation project, requirements for continuity must be examined. The overall program of NSRDS is designed to ensure continuity of effort in the production of data compilations needed by scientists and engineers. In some areas a revised and updated compilation may be needed every six months; in others, only every four or five years. In almost all areas, continuing literature review and indexing operations are required to maintain a current awareness of the state of development of the field. Therefore, most new projects undertaken by NSRDS are expected to be long term, continuing activities, maintained as one component of the normal range of professional activity of the program leader.



Activities and products of data evaluation centers.

The broad types of activities and products that are normally associated with a data center are shown in the figure above. The left-hand column represents activities, while the right-hand column indicates a product that may result from the corresponding activity on the left. Following the initial selection of relevant papers from the literature (an activity basic to all evaluation and compilation projects), a bibliography may be prepared in which the literature to be evaluated is classified into several relatively broad categories. After the initial selection, the papers are indexed; this process consists of assigning a number of key words or symbols to each paper to indicate the data content of the reference. The indexed bibliography resulting from this activity is very useful to many groups of specialists.

In consultations to determine the needs of the technical community of the United States for data compilations of all kinds, the staff of the Office of Standard Reference Data has often been told that an indexed bibliography would satisfy most of the needs of the specialists, since many would prefer to evaluate the data themselves and wish only to avoid the labor of locating the sources of the information. However, this attitude does not prevail among those who need a particular value for a calculation of some kind and are not themselves involved in research in the field.

The next step in the production of a critical compilation is the extraction of data from the literature that has been selected. At this stage an uncritical compilation could be issued, if determined to be useful to the technical community.

The preliminary activities of literature selection, indexing, and extraction of data lead finally to critical evaluation. The product of this work is a critical review of the state of quantitative knowledge in some limited area of a field, or a compilation of critically evaluated data. For NSRDS, a published product must contain sufficient argumentation for the user to know how results were obtained, as well as appropriate reference to the sources of the data used in the final evaluation.

The original emphasis in NSRDS was on the production of critical reviews and data compilations. Experience has shown that other evaluated products are of vital importance as well, and data centers are now encouraged to pay considerable attention of them. Of special significance are "criteria," which may be of three types: (a) criteria for measurement methodology in the laboratory, (b) criteria for reporting results in the literature, and (c) criteria for evaluating results already reported.

The physical form of the products of NSRDS activities may be anything considered convenient by the users to whom the product is directed; that is, the product may be a monograph, looseleaf data sheets, a journal article, microfiche cards, IBM punch cards, punched paper

tape, magnetic tape, or any other physical form in which information may be stored.

The data-systems design and research activity of the Bureau's Office of Standard Reference Data is concerned with the problems of handling data throughout the entire flow process—from the time measurements are first made in the laboratory, through disclosure in some form to persons who may use the results (as a journal article, a laboratory report, or perhaps a magnetic tape), through review, selection, and evaluation procedures in the data center, and finally to dissemination among members of the technical community who have use for the evaluated product. The use of computers for all practical operations is emphasized; such applications to speed the flow of data from the measurer to the user have only just begun.

Program decisions of the Office of Standard Reference Data have relied heavily on the advice of a representative cross-section of the American technical community. As overall program review committee for the work of the Office, the Executive Committee of the Office of Critical Tables of the National Academy of Sciences—National Research Council provides policy recommendations and is an important channel of communication with many segments of the technical community. Program officers in other Government agencies have been consulted to determine the needs of their mission-oriented programs for the products and services that the standard reference data system is intended to provide.

Considerable reliance has been placed on the recommendations of panels of specialists in each of the technical areas where a program is being operated; one or more meetings of ad hoc panels have been held in each area. Some of these panels are existing committees of the NAS—NRC, established primarily for other purposes. Others have been assembled directly at the request of the Bureau. These advisory panels are now being organized on a continuing basis under the auspices of the National Academy of Sciences—National Academy of Engineering.

The NAS—NRC Office of Critical Tables has provided frequent advice on needs, priorities, and other operational details, and has also served as a channel of communication to various segments of the technical community in the United States and abroad. In all, more than 200 leaders of American science and technology have given generously of their time and experience in helping to make NSRDS most responsive to the needs of the technical community.

General Status of the Program

As a result of the recommendations of advisory panels, greatest emphasis has been placed on initiation of new projects for evaluation and compilation and on expansion of old ones, leaving to a future of greater affluence the implementation of extensive and sophisticated

information services. Significant progress has been made, especially in the areas of thermodynamic and transport properties and of atomic and molecular properties; these two categories have been judged to be of highest priority for additional effort. In the field of nuclear data, existing activities sponsored by the U.S. Atomic Energy Commission provide nearly adequate coverage of the technical scope required, although the level of effort needs to be increased to meet the rapid rate of appearance of new data.

More detail on the activities of the Office of Standard Reference Data and on the status of specific projects under the cognizance of this office are described in a recent report by the Office of Standard Reference Data [8].

The Standard Reference Data Act

Initial administrative responsibility for the National Standard Reference Data System was given to the National Bureau of Standards by the FCST. This assignment was made in recognition that many of the broad elements of the proposed national program fell within already-defined authorities which the Bureau had been exercising for some time. However, the early years of operation of the System revealed that certain additional authority from Congress would provide increased efficiency in supplementing the program. Seeking such authority, the Department of Commerce submitted appropriate draft legislation to the 89th Congress. After public hearings before the Subcommittee on Science, Research and Development (House Committee on Science and Astronautics), a revised bill was reported favorably by the full Committee; it was passed by the House of Representatives in mid-August 1966. Although the bill did not move to completion in the 89th Congress, it was resubmitted to the 90th Congress, further revised by floor action in the House of Representatives, passed by the Senate and signed into law on July 11, 1968.

The final version of the legislation contains the following provisions: (i) a declaration that it is the policy of the Congress to make critically evaluated reference data readily available to scientists, engineers, and the general public; (ii) a directive to the Secretary of Commerce to provide or arrange for the collection, compilation, critical evaluation, publication, and dissemination of standard reference data; (iii) a directive to the Secretary of Commerce to prescribe standard criteria and procedures for the preparation and publication of standard reference data, as may be necessary; (iv) authority for the Secretary, or a person or agency designated by him, to sell standard reference data and to allow the proceeds to be used by the Bureau; (v) authority for the Secretary to obtain copyright, on behalf of the United States as author or proprietor, for standard reference data prepared or made available under the Act; and (vi) an authorization for appropriations

to the amount of \$1.86 million for the present fiscal year, and a requirement for annual authorization for subsequent appropriations.

The authorities indicated in items (iv) and (v) above offer NSRDS new or modified channels by which to distribute reference data to users and the potential to use these same channels for recovering a portion of the cost of the program. Study is already underway as to how NSRDS should act to utilize these authorities in the common good.

International Cooperation

Evaluation and compilation of data on the properties of substances has been a joint activity of the world's scientists for many years. The International Critical Tables, produced mainly between 1920 and 1930, contained contributions from scientists all over the world, coordinated through the efforts of the National Academy of Sciences. The tables of Landolt-Börnstein, originally German, now contain contributions by scientists from many countries. Compilation and evaluation of neutron cross section data have become a broad international effort, with participation by centers in the United States, Canada, the United Kingdom, France, the Soviet Union, and other countries.

The establishment of NSRDS in the United States has stimulated additional interest among scientists in other countries on the possibility of developing cooperative programs with scientists in the United States. Possible cooperation has been discussed with scientists from the United Kingdom, France, Germany, the U.S.S.R., and Japan. Such widespread interest leads immediately to the concept of a multilateral international program, incorporating activities from all countries wishing to participate. Indeed, multinational cooperation through several of the international scientific unions has been under way for many years. The International Unions of Pure and Applied Chemistry and Pure and Applied Physics and the International Astronomical Union have been especially active. In June 1966 the International Council of Scientific Unions created a Committee on Data for Science and Technology (now called CODATA) whose function is to promote international cooperation, to serve as a channel of communication among projects in various countries all over the world, to encourage more scientists to undertake projects of this type, and to make recommendations about needs and priorities to persons responsible for funding such projects in the various countries.

The second annual meeting of CODATA was held at the Headquarters of the Soviet Academy of Sciences in Moscow on May 31, and June 1 and 2, 1967, and the third annual meeting in Frankfurt, Germany, June 28 and 29, 1968, both under the chairmanship of Frederick D. Rossini, Vice President for Research at the University of Notre Dame. The Chief of the Office of Standard Reference Data was invited to attend both meetings as an observer and was thereby enabled to learn

about numerous data compilation activities in the U.S.S.R. and other countries.

Membership in CODATA consists of National Members, Union Members, Liaison Representatives, and Co-opted Expert members. National Members, as of July 1968, represent Canada, France, Germany, Japan, United Kingdom, USA, and U.S.S.R. Italy and Poland have indicated their desire to become members. There are eleven Union Members, five Liaison Representatives and one Co-opted Expert Member—Dr. Guy Waddington, Director of the Office of Critical Tables of the United States National Academy of Sciences. The Committee is served by a full-time executive staff under the directorship of Dr. Christoph Schäfer, with a Central Office located in Frankfurt, Germany.

The First International CODATA Conference was held June 30 to July 5, 1968, at Arnoldshain, near Frankfurt, Germany. Approximately 100 scientists and administrators attended the conference, representing twelve countries.

The International Council of Scientific Unions, the unions themselves, and CODATA are all nongovernmental organizations; however, preliminary informal discussions have also been held regarding the desirability and feasibility of conducting an international standard reference data program under the auspices of an intergovernmental organization, such as OECD, or one of the United Nations specialized agencies, or a new agency created for the purpose.

Competence and interest in an international cooperative program for compiling reference data are found in most of the technically developed countries. The products of such a program would benefit any nation that conducts a research and development effort of any size, not merely the most highly developed countries. International cooperation in this area has been a tradition of the world's scientists for at least half a century, but until recently no mechanisms for overall coordination and support have existed. Many arguments now favor the vigorous development of an international cooperative program.

Current Data Project Activity

Accomplishments of the past year, include: a) specific data compilation products published as a result of project efforts, and b) coordination and communication activities involving members of the Office of Standard Reference Data, scientists engaged in data projects, scientific advisors to NSRDS, and representatives of the user community. These accomplishments are described below under the headings by which OSRD classifies its efforts.

Nuclear Properties

The Office continued its direct sponsorship of the Photonuclear Data Center. A supplement to the initial publication of this Center, "The

Photonuclear Data Index and Annotated Bibliography of Photonuclear Cross Sections," was published this year.

The Office has continued fulfilling its responsibilities in this field by providing assistance to appropriate agencies. The program manager in this area is a member of the Cross Section Evaluation Working Group which has prepared a set of evaluated neutron cross sections. In addition, he is a member of the Cross Section Review Committee of the Defense Atomic Support Agency, has been appointed to the American Nuclear Society Standards Committee and is Chairman of its Subcommittee on Nuclear Technology.

The Second Neutron Cross Sections and Technology Conference was held in Washington, March 1968. Sponsored jointly by NBS, AEC, ANS, and APS, the Conference had as its Chairman the OSRD program manager for nuclear data, who is also editor of the proceedings of the Conference.

Atomic and Molecular Properties

The Atomic and Molecular Properties area encompasses primarily property characteristics of individual atoms or molecules rather than of substances in any specific state of aggregation. A more explicit indication of the scope of this area is given in the list below.

Major Topics in Atomic and Molecular Properties

Fundamental constants and properties	Information on molecular energy levels derived from spectral data:
Atomic energy levels	a) diatomic molecules
Atomic spectral data	b) polyatomic molecules
Atomic and molecular x-ray spectral data	Other well-defined properties of atoms and molecules
Atomic and molecular collision data	Transition probabilities
Particle-surface interactions	Computed functions
Plasma properties	Interatomic and intermolecular forces

Reference data in this area find application in two broad categories—basic scientific research and the extension of theoretical understanding on one hand, and technological problems, including analysis and identification of substances, on the other.

General guidance for the Atomic and Molecular Properties program is provided by an advisory panel headed by Dr. E. U. Condon of the University of Colorado. At the outset, the advisory panel recognized that special attention would be necessary in several areas, and that subpanels of specialists for individual topics should appropriately be convened to consider needs.

Four specialized advisory groups have met so far: (a) The continuing advisory body on infrared spectroscopy is the Board of Management of the Coblenz Society. This group has met several times at the request of the Office of Standard Reference Data to provide

advice and guidance of several sorts. One of its specific tasks, to prepare criteria for the evaluation of infrared spectra, has led to a report published in the August 1966 issue of "Analytical Chemistry". In addition, the Board has given advice on the development of infrared spectral data compilation projects at various quality levels. (b) An ad hoc advisory group on NMR spectroscopy, organized by Dr. B. L. Shapiro, met in November 1967 in Washington, D.C., to discuss data needs and possible compilation projects on chemical aspects of NMR spectroscopy. (c) An ad hoc panel on the application of computers to spectral information was convened on July 24 and 25, 1967, with Dr. E. R. Lippincott as Chairman. In addition to recommendations to the Office of Standard Reference Data, this panel provided discussions leading to a U.S. presentation at the 9th European Congress on Molecular Spectroscopy, held under IUPAC sponsorship in Madrid, September 10-15, 1967. (d) An ad hoc panel on Raman spectroscopy, with special concern for techniques involving laser excitation, met in Washington, D.C., on May 24, 1968.

Most of the data projects in the Atomic and Molecular properties area have issued valuable products. During the past year, the following publications have appeared:

Publication date	Title	Author(s)	Publication Number
9/67	Bibliography of Atomic and Molecular Processes for July-December 1965.	L. J. Kieffer-----	ORNL-AMPIC-6.
9/1/67	Selected Values of Electric Dipole Moments for Molecules in the Gas Phase.	R. D. Nelson, Jr., D. R. Lide A. A. Maryott	NSRDS-NBS-10.
9/25/67	X-Ray Wavelengths and X-ray Atomic Energy Levels.	J. A. Bearden-----	NSRDS-NBS-14.
10/67	Tables of Molecular Vibrational Frequencies, Part 2.	T. Shimanouchi---	NSRDS-NBS-11.
11/30/67	Selected Tables of Atomic Spectra: a. Atomic Energy Levels—Second Edition. b. Multiplet Tables. Si I, Data Derived from the Analyses of Optical Spectra.	C. E. Moore-----	NSRDS-NBS-3 section 2.
12/67	Compendium of ab initio Calculations of Molecular Energies and Properties.	M. Krauss-----	NBS Technical Note 438.
3/68	Tables of Molecular Vibrational Frequencies, Part 3.	T. Shimanouchi---	NSRDS-NBS-17.
4/68	Critical Review of Electron Impact Ionization Cross Sections.	B. L. Moiseiwitsch, S. J. Smith	Review of Modern Physics, 40, 238-353 (1968).
6/68	Tables for the Rigid Asymmetric Rotor.	R. H. Schwendeman.	NSRDS-NBS-12.

Thermodynamic and Transport Properties

There are 17 currently active projects in the NSRDS program on Thermodynamics and Transport Properties of which 12 are sponsored by the Office of Standard Reference Data. The topics covered are:

Chemical Thermodynamic Data on Inorganic Compounds.

Thermodynamic Data on Organic Compounds.

Properties of Molten Salts.

Thermodynamic Properties of Ammonia.

Low Temperature Specific Heats.

Thermodynamic Properties of Liquid Metals & Liquid Oxides.

Phase Behavior in Binary and Multicomponent Systems at Elevated Pressures.

Vapor-Liquid Equilibrium in Binary Mixtures of Non-Electrolytes at Low Pressures.

Properties of Electrolyte Solutions.

Thermodynamic Properties of Metals and Alloys.

Binary Metal and Metalloid Constitution Diagrams.

Thermodynamic Functions of Cryogenic Fluids.

Thermal Conductivity of Selected Materials Including the Elements.

Viscosity and Thermal Conductivity of Mixtures in the Gaseous and Liquid States.

Transport Properties of Cryogenic Fluids.

Thermal Conductivity of Solids at Low Temperatures.

The following reports have been published:

Selected Values of Chemical Thermodynamic Properties, Part 3, Tables for the First Thirty-Four Elements in the Standard Order of Arrangement, D. D. Wagman, W. H. Evans, V. B. Parker, I. Halow, S. M. Bailey and R. H. Schumm—NBS Technical Note 270-3, Jan. 1968.

Thermal Conductivity of Selected Materials, Part 2, C. Y. Ho, R. W. Powell and P. E. Liley—NSRDS-NBS-16.

Critical Analysis of the Heat Capacity Data of the Literature and Evaluation of Thermodynamic Properties of Copper, Silver and Gold from 0 to 300 K, G. T. Furukawa, W. G. Saba, M. L. Reilly—NSRDS-NBS-18.

Thermodynamic Properties of Ammonia as an Ideal Gas, L. Haar—NSRDS-NBS-19.

Annual Index of Worldwide Published Thermochemical Studies.—As a part of its continuing program on the collection and evaluation of chemical thermodynamic data, the Thermochemical Data Center at NBS has participated for several years in preparing the Bulletin of Thermodynamics and Thermochemistry. This annual publication, prepared under the auspices of the International Union of Pure and Applied Chemistry, Commission on Thermodynamics and Thermochemistry, contains listings of thermodynamic work in progress in laboratories throughout the world and an index to the published thermodynamic literature of the past year. The current issue of the

Bulletin includes over 2300 references, with about 8000 citations to chemical species in the index.

Solid State Properties

This NSRDS program effort embraces the activities of four data centers covering such areas as crystal properties data, alloy properties data, cryogenic properties data, and diffusion in metals and alloys. These centers compile bibliographies and help stimulate the compilation of data related to their area of interest.

The Alloy Data Center has now developed and made operational a computerized bibliographic system for the storage and retrieval of information. Its description will soon be published. The Crystal Data compilation is now in production with approximately 4000 data entries of Volume I stored on tape for computer-controlled text composition.

Chemical Kinetics

This program area continues to emphasize the preparation and publication of critical review monographs on highly selective subjects. Two manuscripts have been issued and a number are now being processed for publication. The two which have been issued are:

NSRDS-NBS-9, "Tables of Bimolecular Gas Phase Reactions" by A. F. Trotman-Dickenson and G. S. Milne.

NSRDS-NBS-13, "Catalytic Hydrogenation of Ethylene on Metallic Catalysts" by J. Horiuti and K. Miyahara.

A third publication, "A Method for Estimating the Arrhenius A Factors for Four and Six-Center Unimolecular Reactions" by H. E. O'Neal and S. W. Benson has appeared in the Journal of Physical Chemistry and is the byproduct of an intensive study on the kinetics of gas phase unimolecular reactions. The effort concerning the evaluation and tabulation of reaction rate data has been enlarged to include data sheet preparation on the kinetics of those reactions considered important to related fields such as aerospace and air pollution.

Two data centers continue to perform a valuable service in compiling and disseminating information in chemical kinetics and radiation chemistry. The center treating the latter has compiled nine bibliographies in response to author requests and is stimulating the preparation of a data sheet on the decomposition of methane. It also publishes a weekly reading list of articles in the field of radiation chemistry appearing in the current literature.

The Chemical Kinetics Data Center plans to issue a bibliography of kinetic data on reactions of nitrogen, oxygen, and the nitrogen oxides as well as a compendium of rate coefficients of elementary reactions in the light elements.

Colloid and Surface Properties

There are currently six active projects in this area under OSRD sponsorship. The fields covered are the following:

Critical Micelle Concentrations of Association Colloids.

Electrical Properties of Interfaces.

Surface Tension of Molten Salts.

Light Scattering by Liquids.

Surface Tension Data of Pure Liquids.

Physical Data Pertaining to Phase Transition Kinetics.

In addition, there are three projects being carried out under the auspices of the NAS-NRC Committee on Colloid and Surface Chemistry which will lead to critically evaluated data on heats of immersion, properties of monolayers, and surface tensions of colloidal solutions. A report on the surface tension of molten salts is in the final stages of review and editing.

Data Systems Design and Development

The Data Systems Design and Development group (formerly called Information Systems Design and Research) combines interest in computer programming, systems design and application, and data correlation. Earlier experience gained in design and application of a number of general purpose computing programs (like OMNITAB) is now providing guidelines for the preparation of general-purpose computer programs for data storage and retrieval, for file manipulation, and for computer-assisted text preparation, editing, and printing. There is close collaboration with the NBS Electronic Printing Section in devising a variety of computer-based systems for more effective use of the Electronic Composing System at the Government Printing Office.

Work in this area can be grouped into three main activities:

(a) development of general-purpose computer programs required for the generation and updating of data files and the preparation of publications.

(b) design of computer-based data files and retrieval systems which provide accessibility to as wide a segment of the technical community as possible.

(c) consultation and advisory services to data centers adhering to NSRDS.

Computer-Assisted Text Preparation and Editing.—Special effort is being given to the writing of general-purpose compatible computer programs to exploit the capabilities of the newest computer configurations. Programs and the subroutines they require are being written in ASA Standard FORTRAN IV. The first release in this series is EDPAC. It contains five programs that have been used extensively at NBS over the last two years. These are called JUSTIFY, SCRAMBLE, SEARCH, BLOCKSEARCH, and SUBSTITUTE.

The characteristics of EDPAC were outlined in some detail in the *NSRDS Newsletter* of December 1967.

Automatic Typesetting and Composition of Data Tables.—Experience has been gained in writing computer programs to convert existing data tapes for automatic typesetting of NSRDS publications. One goal has been to design the programs, insofar as possible, for general rather than specific applications.

Computerized Data Files.—The design and development of an experimental computerized file of Standard Reference Data for remote access via teletype has been undertaken. This work will draw on experience with a variety of time-shared computer systems. Efforts are being directed toward arranging the subject matter to make files attractive to university computing centers and commercial groups offering time-shared computer service to schools and colleges. Plans are to make the subject matter of general use to teachers of physics and chemistry.

Information Services

Because the major available resources of the NSRDS program have been applied to the production of compilations and critical reviews, the development of a vigorous Information Service activity has been delayed. Nevertheless, groundwork for services has been laid and pilot experience is being acquired. Four basic units of activity for Information Services have been identified: (a) Data File, (b) Compilation Publication Services, (c) Inquiry Services, and (d) Analysis and User Relations.

Data File.—The task of this activity has been to acquire known world-wide data compilations and to organize the collection systematically. The File houses a collection of about 1000 reference data compilations, one of the most extensive collections in the world. General planning of ultimate methods of indexing, storage, and retrieval has begun.

Compilation Publication Services.—This activity acts as an editorial intermediary between OSRD, associated data centers, other individuals and groups producing data compilations on the one hand, and publishers on the other hand, including the Government Printing Office. In the present reporting period, 13 publications have been produced and 20 are in process.

Inquiry Services.—Plans have been developed for systematic handling of requests for data which may be addressed to any part of the National Standard Reference Data System. Under these plans, replies will be obtained by using all of the System's resources—the Data File, the associated data centers, and experts in NBS. At the present time

only limited replies can be provided—referral to a data center or to a specialist, provision of reference sources, or provision of a pertinent data compilation or excerpt therefrom are the most common responses. More than 400 inquiries were handled in the past year, and only 28 responses were negative because of lack of information or the unavailability of a likely source of information.

Analysis and User Relations.—The mission of this unit is to provide OSRD with an understanding of requirements, both present and future, of NSRDS actual and potential users. It also has the responsibility for providing information on how best to meet user needs and to develop feedback mechanisms for evaluating services provided. The unit has a pilot study underway aimed at determining the needs of scientists in finding and using data.

NSRDS News.—This newsletter first appears as a special section of the monthly *NBS Technical News Bulletin* and then is reprinted separately and sent to more than 3200 interested individuals. Regular features include an updated list of publications from the National Standard Reference Data System, descriptions of data centers and compilation projects, and other items of news.

THE STANDARD REFERENCE MATERIALS PROGRAM

For more than 60 years, in an era marked by rapid and sweeping technological change, certified Standard Reference Materials (SRM) from the National Bureau of Standards have played a pivotal role in providing a basis for order, stability, and control of the productive process. In this time the gross national product has multiplied nearly 20-fold, from around \$45 billion to some \$850 billion. A high-technology civilization has developed, based in large part on the mass production of goods and services, with requirements for ever-increasing accuracy of measurement, interchangeability of parts, and with stringent performance criteria for materials of all types. Standards to establish and maintain these criteria form the basis of this technological process, and NBS Standard Reference Materials stand in the forefront of the thousands of standards produced and certified throughout the world.

History of the Program

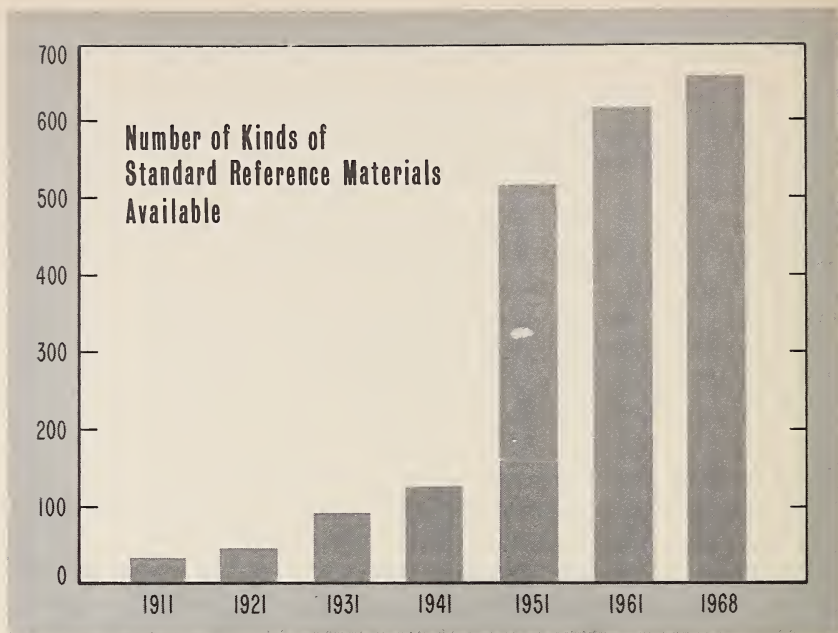
The Bureau's Standard Reference Materials program began in 1906 when four standard samples, as they were then called, were produced and certified to help control the quality of cast iron in the U.S.A. With this start the United States thus became the first country to have a national standard materials program. Today, this program is being significantly broadened in line with current needs of science and industry as shown by the more than 650 different SRM's now produced in more than 70 different categories.

Both industry and Government, in quest of better quality control, smoother exchange of goods in commerce, clearer definition of performance characteristics, and more comprehensive measurement competences, rely on NBS Standard Reference Materials as crucial reference points in the establishment of an effective measurement system for the entire Nation. Through the use of the Bureau's certified SRM's, science and industry can make onsite calibrations and evaluations of measuring instruments, methods, and systems in their own laboratories and plants.

The NBS Office of Standard Reference Materials conducts its program on a self-supporting basis with the full cooperation of industry. Outputs are increasingly in demand in such fields as metals, minerals, chemicals, polymers, biological materials, gases, radioisotopes, ores, and ceramics. Among leading customers for some of the 650 currently available SRM's are companies and organizations concerned with aerospace, oceanography, pollution control, nuclear energy, clinical medicine, and such industries as steel, brass, aluminum, rubber, glass, cement, petroleum, automotive, chemical, plastic, pharmaceutical, and transportation.

In this day essentially all production is controlled by, and the quality of output checked through, highly sophisticated instrumentation. Calibration of this equipment so that readings in widely separated plants and between producer and customer may be referred to a common, accepted base is one of the large roles played by SRM's. It is because the properties of NBS—SRM's are, in general, certified on an absolute basis that they are widely accepted as the primary standards with which the intercalibration process may be undertaken with confidence. Instruments calibrated range from pH meters costing \$100 or less to automatic reading emission spectrometers ranging in cost upwards of \$75,000. Physical properties measured and certified range from micron-sized particle distributions, through density, viscosity, dielectric properties, and Mössbauer effect, to extremely complex property certification such as thermal emittance. Certification for chemical composition ranges from a steel SRM where only the carbon content is certified, to a very high purity zinc where more than 20 elements have been measured, some at the parts per billion level.

As America's continuing technological revolution proceeds unabated, pressing demands for new Standard Reference Materials increase, exceeding resources currently available for their certification. In meeting its responsibilities, therefore, the Office of Standard Reference Materials necessarily exercises a continuous process of judgment in deciding which new materials should be chosen for preparation and in what order of priority, so as to serve the best overall interests of the Nation's science and technology. This task is done only after exten-



sive consultation with various standardizing bodies, industry representatives, and other interested groups.

The total number of standard materials used by science and industry is far greater than the approximately 650 SRM's produced and distributed by the Bureau, but these and the 120 now in production have been selected for development on the basis of priority for purposes of technological progress. The NBS Standard Reference Materials serve, in fact, as essential benchmarks to which secondary industrial standard materials are related. In establishing the priority for the production of a new Standard Reference Material, particular emphasis is given—

- where attainment of needed accuracy of analysis or measurement of characteristics is not economically or technically feasible elsewhere,
- where industry-wide standards for commerce are needed from a neutral supplier not otherwise available,
- where continuing availability of highly characterized material from a common source is important to science or industry.

How the Standard Reference Materials program has both expanded and changed with the times is apparent not only in statistics of the program's growth but also in the extraordinarily demanding nature of current applications of high accuracy new Standard Reference Materials.

In 1910, for example, standards for a half a dozen elements certified to a fraction of a percent were good enough for steel industry needs

of the time. Now, in accounting for the uranium content in fuel rods used in nuclear power plants it is necessary to have a uranium assay standard certified to 1 part in 10,000. Similar highly accurate SRM's are currently available from NBS.

Acute needs have developed for new Standard Reference Materials useful in determining trace levels of many elements, particularly in high-purity materials such as are used in the transistor and semiconductor industries. The nature of current requirements is illustrated by the case of high-purity silicon, for which certification of elements will be needed at or near the level of one part in a billion.

In order to measure trace elements at these extremely low levels, instruments like the spark-source solids mass spectrograph and techniques such as neutron activation analysis have been developed. While such instruments and techniques are extremely sensitive in detecting trace elements at the part per billion level, they are often not very accurate, since until recently SRM's were not available to ferret out sources of systematic error. Systematic errors of 300 percent, common as little as 2 to 3 years ago in the use of the spark-source mass spectrograph at the part per million (and lower) level have now been reduced by a factor of 10 or more. One of the factors contributing to this advance has been the issuance of high-purity platinum, gold,



NBS Standard Reference Materials (foreground) are used to analyze alloy samples on an x-ray fluorescence device. Photograph furnished through the courtesy of the Research Laboratories of General Motors Corporation.

zinc, and other similar SRM's, where elements at these very low levels have been measured and certified at NBS. The application of these SRM's to activation analysis and other sensitive techniques will have a similar impact.

Standard Reference Materials contribute to progress in every arena of action from the reaches of space to the depths of the ocean, enhancing the quality of life for Americans at work and at play, in factory and field, in sickness and in health.

Recently, for example:

- At the request of the College of American Pathologists and the American Association of Clinical Chemists, the Bureau prepared a Standard Reference Material for cholesterol in human blood, helping to correct a situation in which clinical determinations could vary by as much as 25 to 50 percent. This SRM is particularly important in research on the causes and prevention of atherosclerosis.
- For widespread applications in medical research and treatment, NBS has prepared iodine and sodium tracers of known radioactivity.
- To provide reliable analytical methods and standards for purposes of monitoring pollutants under the Clean Air Act, the Bureau produced Standard Reference Materials for determining amounts of sulfur in various fuel oils.
- NBS-produced standards for detecting long term changes in the concentration of carbon dioxide in air (having implications in long range weather changes) are used by the Environmental Science Services Administration to calibrate monitoring equipment ensuring that measurements taken over many years can be referred to a common base.
- The Bureau's reference materials for metallo-organics in oil are used by the Departments of Defense and Transportation and by industry to determine engine wear and overhaul needs in trucks, trains, and planes, allowing for efficient and safe overhaul schedules. By this means, catastrophic failures are averted, lives are saved, and maintenance costs reduced.
- U.S. Customs laboratories check their apparatus against the NBS sucrose standard in order to levy uniform duties on imported sugar, whose annual volume amounts to some \$500 million.
- Transfer of half-a-billion dollars worth of nuclear fuel from the Atomic Energy Commission to private industry over a two-year period was based on NBS assay standards. The international control and transfer of fissionable uranium and plutonium are dependent on these standards. These SRM's are vital in the area of nuclear non-proliferation control.

- Microfilm reproduction systems—a business grossing at least \$300 million a year—benefit from microcopy resolution test charts developed by the Bureau for evaluation purposes. The Department of Defense and the General Services Administration use these standards as a basis for a large portion of their contract specifications.
- Consumer-goods producers of all kinds, particularly in the garment and textile fields, look to the NBS unit known as the “standard fading hour” as a means of putting all fading measurements on a common base. To extend the competence into the Nation’s laboratories, the Bureau developed standard light-sensitive paper and light-sensitive plastic chips. The chips serve to produce controlled fading at a much accelerated rate, and thus facilitate more rapid tests under simulated natural conditions.
- Nuclear instrument manufacturers have begun to utilize three new carbon-14 labeled solution SRM’s for liquid scintillation counting in the standardization and calibration of nuclear instruments. The new standards also are important to research workers concerned with accurate measurements of low-energy beta emitters, and are widely used in medical research.
- In cooperation with the electrical, electronics, and aerospace industries, the Bureau has certified six new beryllium-copper alloy SRM’s for calibrating optical emission and x-ray spectroscopic methods, to help meet critical needs for analytical reference standards for these widely used alloys. Almost every high-reliability military, space, and oceanographic system uses beryllium-copper alloys in specialized electronics equipment and as components of microminiaturized circuitry.
- A new zinc freezing-point Standard Reference Material with a purity approaching 99.9999 percent has been certified by NBS. The General Conference on Weights and Measures has placed the freezing point of zinc on a par with the boiling point of sulfur as a fixed point of the International Practical Temperature Scale. The zinc point is expected eventually to replace the sulfur point on the Scale.

It is readily apparent from the summary in table 1 that the program in past years has strongly supported heavy industry—steel, copper, rubber, etc. This is simply a consequence of the historical development of this program over the years. However, as rapidly as funds and resources become available, the SRM program is expanding to meet national needs in health, air and water pollution, resource development and in other critical areas. Experts in each of these fields have decried the lack of standards and have emphasized that progress will necessarily be slow until these needs are met.

Additional support from other interested Government agencies having direct missions in the fields of health and pollution control, if

TABLE 1. *Present SRM Inventory*

Category	No. of SRM's available	Typical use
1. Metals, Ferrous-----	171	Quality control-steel industry.
2. Metals, Nonferrous-----	101	Be-Cu alloy for aerospace, oceanography.
3. Ores, Cements, Ceramics-----	36	Control of fineness of cements.
4. Organics, Organo-metallics-----	84	Cholesterol for clinical control.
5. Primary SRM's for pH, acidimetry, gases, high- purity metals.	35	Trace element determinations in platinum, gold, zinc by advanced techniques.
6. Nuclear, Isotopic, Radio- activity.	68	Determination of geologic ages by Pb isotopes.
7. SRM's for Thermodynamic Properties.	13	Calibration of resistance ther- mometers.
8. SRM's for Rubbers, Plastics, Polymers.	22	Molecular weight determina- tions.
9. SRM's for Various Physical Properties (e.g., viscosity, thermal emittance).	87	Control of viscosity of glasses.
10. SRM's for Color Specification and Control.	50	International color system.
11. Miscellaneous-----	2	Specifications for microcopying.
Total-----	669	

TABLE 2. *Work in process (1/68)*

National concern area	Percent of total	Thousands of dollars invested
1. Conservation and Resource Development-----	6. 0	\$92
2. Defense-----	18. 2	281
3. Health-----	4. 4	68
4. Education (Scientific Advance)-----	23. 5	363
5. Pollution-----	1. 3	20
6. Economic Growth-----	24. 9	385
7. Safety-----	4. 8	74
8. Space-----	12. 5	193
9. Consumer Interests-----	3. 0	46
10. Government Operations-----	1. 4	22
Total-----	100. 0	1, 544

forthcoming, will allow significant increases in the production and certification of SRM's to support these areas.

Similar contributions by the NBS Office of Standard Reference Materials are planned for other vital applications, including:

- Standard Reference Materials to be certified in the range of 100 parts per billion for trace metallic elements in blood, tissue, and botanical matter, as related to such life processes as enzyme catalysis, metabolism, reproduction, cell growth, and aging—for use by specialists in fields ranging from medicine to the food industry.
- Preparation and calibration of gas SRM's for sulfur dioxide, hydrocarbons, and carbon monoxide in air to facilitate measurements of some of the most important and highly significant pollutants in the atmosphere.

- Soda-lime glass SRM's representative of the chemical compositions of more than 90 percent of the flat glass and container glass being manufactured; a series of glass SRM's with trace impurities graded at the 500, 50, 1, and 0.02 ppm as well as a "base level blank." Also clay, plastic, and flint SRM's—all selected in cooperation with ceramics and glass industry representatives to meet urgent research and production control needs.

Current Activity

The past year has been one of continuing growth in the SRM program, reflecting the increased capitalization in support of the work. Over 43,000* units were sold to over 4,500 customers both in the United States and throughout the world. Approximately 25 percent of sales are to foreign customers indicating the international importance of the program. The dollar value of total sales for FY 68 was \$1,022,500. Table 3 shows how these sales were distributed among the major categories of the SRM inventory.

TABLE 3. *Fiscal year 68 Sales (By Major Category)*

Category	Units sold	Sales value in thousands of dollars	Per-cent of total sales
1. Metals, Ferrous.....	17, 543	\$438. 1	42. 8
2. Metals, Nonferrous.....	3, 164	102. 7	10. 0
3. Ores, Cements, Ceramics.....	2, 812	46. 3	4. 5
4. Organics, Organo-Metallics.....	3, 019	65. 5	6. 4
5. Primary SRM's: pH, acidimetry, gases, high purity metals.	5, 122	69. 6	6. 8
6. Nuclear, Isotopic, Radioactivity.....	1, 225	62. 0	6. 1
7. SRM's for Thermodynamic Properties.....	991	14. 9	1. 5
8. SRM's for Rubbers, Plastics, Polymers.....	4, 173	76. 8	7. 5
9. SRM's for Various Physical Properties.....	3, 341	72. 8	7. 2
10. SRM's for Color Specification and Control.....	1, 206	27. 6	2. 7
11. Miscellaneous.....	1, 667	46. 2	4. 5
Total.....	43, 263	1, 022. 5	100. 0

During this reporting period 112 new and renewal SRM's were produced, certified, and placed in stock. The 120 projects now in progress will produce an additional 268 new and renewal SRM's. The following pages give in detail the technical story of a representative sampling of some of these new SRM's.

In a continuing assessment of the needs for SRM's throughout the Nation and the world, in FY 66 the Office of Standard Reference Mate-

*Equivalent to 70,000 units on the old reporting base. The previous reporting method distorts the average picture by overemphasizing unit sales of the micro-copy test charts.

rials identified 167 SRM's required which would cost \$9,060,000 to produce. In FY 67 and 68, the corresponding figures are 226 SRM's at a cost of \$12,328,000 and 251 SRM's at a cost of \$15,084,000. Present indications show little or no signs of leveling in demand for new SRM's; indeed, as standards in the fields of health, air and water pollution, and other relatively new areas are further identified, it is expected that this demand will move upward explosively.

The following examples have been selected from among the 112 new or renewal SRM's produced and certified during FY 68. The technical work, spread across 13 technical divisions of the Bureau, ranged from fairly simple chemical analyses to highly sophisticated and complex physical and chemical measurements. A great deal of this work is reported in detail in the NBS technical literature and in various journals which are referenced in the appendix.

Certified Purity Cholesterol: SRM 911.—At the request of the College of American Pathologists and the American Association of Clinical Chemists, the Office of Standard Reference Materials is now issuing cholesterol of certified purity (purity 99.4 ± 0.3 percent) as an NBS Standard Reference Material for use in standardizing clinical determinations of cholesterol.

The purity of this cholesterol, estimated with 95 percent confidence, has been determined by a combination of gas-liquid chromatography, thin-layer chromatography, and mass spectrometry. The requirements of the Cholesterol Certification Program of the College of American Pathologists were utilized in certifying this cholesterol. These include purification by the dibromide method; determination of melting point and carbon-hydrogen content; suitable reproducibility of results using the Liebermann-Burchard technique; examination by gas chromatography, thin-layer chromatography, and mass, infrared, and nuclear magnetic resonance spectroscopy; and testing for the presence of lathosterol.

There is considerable uncertainty regarding the shelf-life of cholesterol. Therefore, the Bureau will continue to examine the stability of this material over a 2-year period. Any significant changes in purity that may appear will be reported to users.

Platinum Standards: SRM 680 and 681.—Two new platinum standard reference materials, one of high purity and the other doped with impurity elements at the 5 to 10 ppm level, have recently been issued by the Bureau.

These standards are available in wire form. Doping was achieved with a dozen impurity elements at the 5 to 10 ppm (by weight) concentration level. Both standards are certified for silver, gold, copper, iron, iridium, nickel, magnesium, oxygen, lead, palladium, rhenium, and zirconium. In general, values range from a fraction of a part

per million in the high-purity platinum standard to about 10 ppm in the doped standard.

Both standards have been extensively tested and found satisfactory with respect to homogeneity. The following methods were used: optical emission spectrography; spark-source mass spectrography; and electrical measurements, including electromotive force, temperature coefficient of resistivity, and residual resistivity ratio. The recommended values of the concentrations of the various elements given on the provisional certificate were arrived at after analysis by one or more of the following methods: optical emission spectrography, spark-source mass spectrography (isotope dilution), polarography, spectrophotometry, nuclear activation analysis, and vacuum fusion.

Sulfur in Residual Fuel Oil: SRM 1621 and 1622.—Of the sulfur dioxide emitted to the air over the United States in 1963 (greater than 23 million tons), about two-thirds stemmed from coal-burning power plants and another 16 percent from residual fuel oil. The Petroleum Products Survey of the U.S. Bureau of Mines indicates that No. 6 fuel oil sold in the populous Northeastern area ranges from 0.52 percent to 2.9 percent in sulfur content. In the West, the maximum sulfur content exceeds 4 percent.

To provide a base for accurate analysis of fuel oil, NBS has certified two new standard reference materials intended for use in the determination of sulfur in residual fuel oil.

Sulfur was determined gravimetrically as barium sulfate after combustion in a Parr oxygen bomb using 1-gram samples. The method used is similar to ASTM Method Designation D-129. It differed only in that any iron present was removed with ammonium hydroxide before the precipitation of the sulfur as barium sulfate.

Standard Light-Sensitive Papers: SRM 700b and 701b.—Standard light-sensitive paper and booklets of faded strips of this paper are now available for use in standardizing the dosage of the radiant energy from commercial carbon-arc lamps. The standards in the 701b booklet have been faded by exposure to the NBS master lamp in amounts corresponding to stated numbers of "standard fading hours" of exposure between 7 and 25. The standards were calibrated at NBS and the paper from which the standards were prepared was made in the Bureau's paper mill, and was dyed with the direct azo dye, Benzo Azurine G, Color Index No. 24140.

The use of the light-sensitive paper standard is intended to correct for moderate variations in radiant flux. Given the same electrical operating conditions, and similar carbons, the radiant flux emitted by the carbon-arc fading lamp is assumed to be reasonably well controlled with respect to spectral distribution. By using NBS Standard Nos. 700b and 701b it is possible to translate clock hours of exposure in lamps to "standard fading hours" (SFH) based on the average results



Some of the many available **Standard Reference Materials** and associated publications.

of 130 exposure tests using different carbon-arc lamps. Thus it is possible to express the dosage of radiant energy on a common basis in different laboratories.

Turbidimetric and Fineness Standard: SRM 114L.—A renewal of the NBS Portland Cement, turbidimetric and fineness standard, designated NBS Standard No. 114L, has been prepared and certified. This standard may be used to calibrate such devices as the Blaine Air Permeability Apparatus according to Federal Test Method Standard 158, Method 2101, or ASTM Designation C204, and the Wagner Turbidimeter according to ASTM Designation C115. It may also be used to determine sieve residue with a No. 325 sieve according to ASTM Designation C430. These tests are used throughout the cement industry in the U.S. and abroad to control production of cement to specification requirements. Many other uses are developing as the value of particle size control is recognized in a given industry or application.

First issued about 35 years ago, more than a quarter of a million units have since been distributed: No. 114L is the fourteenth renewal of this standard. The standard is now supplemented with calibrated glass sphere standards which extend the particle size range for which NBS standards are available.

Isotopic Standards for Lead: SRM 981-983.—Lead isotopic meas-

urements are used in measuring the age of rocks, meteorites, the earth, and mineral deposits—the latter often of great economic importance. Knowledge of the relationship of meteorites to the earth and understanding of the geologic origins of mineral deposits, can be gained by the measurement of isotope ratios with accurate mass-spectrometric techniques. To advance knowledge of the nature of geological processes such as volcanic action, mountain formation, ore deposition, and the chemical composition of the earth's interior, accurate comparison of data from different laboratories is necessary. This requires standards for calibrating mass spectrometers, now used extensively in this important work, so that lead-isotope data can be compared on an absolute basis.

Three new lead isotope reference standards, designated NBS Nos. 981, 982, and 983, have been prepared. They will allow laboratories to calibrate instruments over the range of isotopic compositions encountered in nature, and to study the performance of their instruments. They are also useful as "spiking" additions for determining lead, as well as for calibrating mass spectrometers.

NBS Microcopy Resolution Test Charts: SRM 1010.—Since December 1, 1967, the microcopy resolution test charts issued by NBS have higher spatial frequencies than those issued previously. The old type was designed in 1963 and had 21 patterns of black bars on a white background ranging from 1 to 10 cycles/mm. These patterns were arranged to permit the extension of the chart to include higher spatial frequencies. The new type has five additional patterns with spatial frequencies of 11, 12.5, 14, 16, and 18 cycles/mm.

American industry and Government spend about a third of a billion dollars annually to microfilm records and preserve films. To assure that the microfilmed images are of adequate quality to store the required information, microfilm contracts generally stipulate that the resolving power of the complete microfilming system be evaluated by means of NBS microcopy resolution test charts. The charts are placed in several locations on the camera copy board and are photographed in the same manner as a document to be recorded. The resulting image is then examined with a microscope. The number of cycles/mm in the smallest pattern in which the bars can be counted with reasonable assurance is the resolving power of the system.

Oxygen-in-Nitrogen Standards: SRM 1604-1609.—The NBS Office of Standard Reference Materials prepared a new series of gaseous standards containing concentrations of oxygen in nitrogen over the range from about 1 ppm of oxygen up to the level of oxygen in the atmosphere.

The new standards are supplied in units of 68 liters at a pressure of 500 psi in disposable gas cylinders. The preparation and certification of the oxygen-in-nitrogen standards required the development of new and

modified analytical methods for determining oxygen in gases. These methods made possible the certification of the oxygen contents of the standards to one relative percent or better at all concentration levels.

Standard Reference Metallo-Organic Compounds.—A series of metallo-organic standards were developed at the request of the Division of Refining of the American Petroleum Institute. At first, the principal users of the metallo-organic standards were the railroad and trucking industries. Here the analysis for metals in lubricating oils was used as the basis for maintenance schedules and predictions of equipment failure. Agencies of the Department of Defense also utilize the metallo-organics for safeguarding equipment and for minimizing maintenance and repair costs. Newer uses include monitoring the presence of catalyst metals and catalyst poisons in process streams.

The amount of wear and the probability of failure of an internal combustion engine may be determined by analyzing its lubricating oil for metals. Such analysis is done with the optical emission spectrometer. Because accurate predictions ordinarily are made on the basis of trends in data accumulated over a period of time from a series of determinations, it is necessary to have standard reference materials for calibrating the apparatus. These standards must be stable, oil-soluble to the concentrations needed, and must not absorb an excessive amount of water. They should yield solutions in lubricating oil which are constant and which will not precipitate on standing.

The certificate furnished with each SRM gives the amount of the metallic element of interest that is present, and also provides directions for preparing a solution of known concentration in lubricating oil.

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INSTITUTE FOR BASIC STANDARDS

The Institute for Basic Standards (IBS), one of three institutes which comprise the National Bureau of Standards, has as its first responsibility the provision of "the central national basis for a complete, consistent system of physical measurement properly coordinated with those of other nations." As a second responsibility IBS develops and maintains standards for physical quantities and for the measurement of physical properties. In concert with the Bureau's Institute for Materials Research, and the Center for Radiation Research, IBS shares the responsibility for providing physical data on the properties of matter and materials.

Implicit in the assignment of the first responsibility is the recognition that there does exist a national system of measurement and that this system is a centralized one, with a central laboratory which develops and maintains the national standards for physical measurement and provides the starting point for a chain of measurement leading from those standards to the ultimate users of the system. This chain must provide for measurements of all necessary magnitudes, from the properties of atoms to those of the universe.

From the point of view of the ultimate user who faces a measurement problem, such as finding the diameter of a ball bearing or the melting point of a metal, the measurement chain can operate in two different ways: (i) It can provide the user with a proven measurement technique or with a calibrated instrument, traceable back to the national standards, with which he can measure the diameter or the melting temperature. (ii) In the case of the melting temperature or other similar properties, it can provide him with an immediately available answer in the form of critically evaluated data which previous investigators have obtained in measurements based on the national standards.

As the Nation's central measurement laboratory, NBS exercises leadership in both these measurement areas. In the Bureau's laboratories the acquisition of standard reference data by precise measurement goes on side by side with research to develop and improve the national standards and associated measurement methods.

PHYSICAL QUANTITIES

The strength and utility of the national measurement system depend fundamentally upon the existence of a complete, consistent system of units and standards around which the system can develop. The In-

ternational System of Units (SI), defined by the 11th and 12th General Conferences on Weights and Measures, is the base for the international system of measurement and for most national systems. Six of these SI units—the kilogram, meter, second, degree Kelvin, ampere, and candela—are the arbitrarily chosen values of six quantities of the physical world—mass, length, time, temperature, electric current, and luminous intensity. Consistent units for other quantities may be derived from these, with appropriate values fixed by the units selected for the basic six. The English system—pound, inch, second, degree Fahrenheit, etc.—and other systems of units are related to the SI units by definite conversion factors.

The research at NBS on physical quantities is concerned with the establishment of these units by international agreement, the realization of the standards which represent them, and the development of a chain of measurement from these standards to the multiples and submultiples needed by our technologically based society. These activities offer an exciting field of technical endeavor which reaches to the frontiers of science and technology. Indeed, the state of sophistication of the U.S. national measurement system is an important gauge of the scope and utility of our science and technology. The results of some of the work in this area are described below; part of the effort involves a collaboration with other groups, and is described in the *Technical Assistance to Others* section.

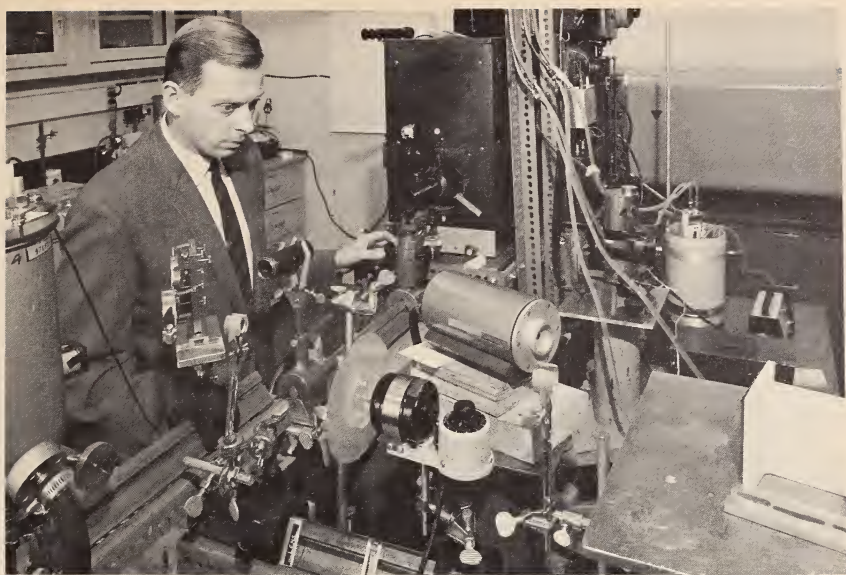
International Base Units

Length

International Comparison of Laser Wavelengths.—A joint report on independent measurements performed at the National Physical Laboratory, Physikalsich-Technische Bundesanstalt, and NBS on commercially available wavelength stabilized helium-neon lasers was prepared and published. The test results showed that unless a specific laser is calibrated, the uncertainty in the wavelength is at least ± 1 part in 10^7 . Agreement on a given stabilized laser can be within ± 5 parts in 10^9 .

Laser Length Standard.—The effect on laser output at $3.39\ \mu\text{m}$ of placing a sample of low-pressure methane inside the laser cavity has been investigated. Saturated absorption resulting in increased laser output power occurs when the laser is tuned to the center of the Doppler-broadened methane absorption line. A linewidth for the emission peak in the laser output of 2 parts in 10^9 was obtained which is a step toward demonstrating the feasibility of this technique as a highly accurate standard of length.

Laser Action on Non-Lasing Transitions in an Atom.—The exact frequency at which lasing action takes place in a gas laser has been



NBS apparatus used for recent international comparison of laser wave-length.

shown to modify the frequency distribution in other, non-lasing lines in the same laser. The detailed analysis of this phenomenon and the phase coherence of a series of cascading transitions predicted an asymmetrical effect on the line shape of the cascading lines. Experiments have been performed to demonstrate this asymmetry.

Time and Frequency

Atomic Second Adopted as International Unit of Time.—A new definition of the international unit of time, the *second*, was adopted Friday, October 13, 1967, in Paris by the 13th General Conference on Weights and Measures. The second has now been defined in terms of a characteristic rate of electromagnetic oscillation of the cesium-133 atom.

Speaking for the governments represented, which include those of all the leading scientific and industrial countries, the Conference agreed overwhelmingly that the moment had come to replace the existing definition, based on the earth's orbital motion around the sun, by an "atomic definition."

The Conference decided that:

The unit of time of the International System of Units is the second, defined in the following terms: "The second is the duration of 9,192,631,770 periods of the radiation corresponding to the transition between the two hyperfine levels of the fundamental state of the atom of cesium-133."

and abrogated the resolutions giving the earlier definition.

Time and Frequency Services.—Standard frequency and time broadcasts continued at VLF, LF, and HF from WWVL, WWVB, and WWV in Ft. Collins, Colo. and at HF from WWVH in Maui, Hawaii. Antenna efficiency for WWVL was increased 30 percent by an improved ground system. Radiated power from WWVH to the Far East was increased with a corresponding decrease in interference on the U.S. mainland by installing reflectors to modify the radiation pattern.

Frequency-Time Dissemination Research.—Time and frequency have the unique characteristic that NBS can distribute them to users by means of standard radio broadcasts. To meet the increasing needs for accuracy in the methods of dissemination, research is underway on new and improved methods. One potential time distribution system being investigated is the use of satellites. Using the VHF transponder aboard the ATS-1 satellite, clocks at NBS (Boulder, Colo.), and NASA (Mojave, Calif.), Anchorage, Alaska, and Pitcairn Island in the South Pacific were compared with an accuracy of better than 10 μ s. In other experiments a clock at NBS (Boulder, Colo.) was compared with a clock at Goldstone, Calif., to an accuracy of about 5 μ s by using the moon-bounce radar system developed by Jet Propulsion Laboratories.

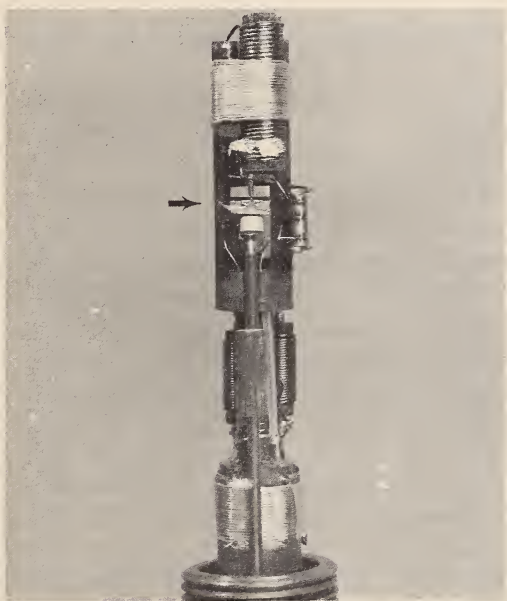
Improvement of the NBS Frequency Standard.—Design and most of the construction have been completed for major modifications to the present NBS Frequency Standard. It is expected to have an accuracy of about 5×10^{-13} and a precision of $1-2 \times 10^{-13}$ for 1 s averaging times. The design makes use of computer-optimized beam optics.

Improvement of the NBS Frequency Standard.—In order to exploit effectively certain ideas relating to the stability of atomic frequency standards, NBS and the Hewlett-Packard Company are jointly developing a new cesium beam tube. The design, which is based on the use of computer-optimized beam optics, and most of the construction have been completed.

Flicker Noise Sources in Electronic Devices.—A system has been developed and tested for measuring the degradation in short-term frequency stability of a signal processed by various electronic devices. Initial tests established the present state of the art for low noise, solid state devices and indicated that near the signal carrier, flicker noise rather than normal thermal noise is the limiting behavior. The use of this test system in the development of new signal processing devices indicates conclusively that a 25 to 35 dB reduction in the flicker noise level of conventional devices is possible by the use of relatively simple design criteria. This reduction has been achieved in the construction of two frequency multiplier chains to be used with the NBS Frequency Standard.

Temperature

Josephson Effect Thermometer.—The need for accurate temperature measurements in the 0.001 to 10 K region has been increasing steadily. Since traditional methods are difficult to use, especially below 1 K, new concepts are being considered, one of which offers the promise of a direct measure of absolute temperature without a calibration comparison. The concept is based on the measurement of thermal noise detected with the aid of the Josephson effect, which occurs when two superconductors are connected by a weak junction. Preliminary experimental results have demonstrated the feasibility of this type of thermometer.



Device being investigated for absolute temperature measurement in the 0.001 to 10 K region. The arrangement of wires on the rectangular metal block is the adjustable Josephson junction (arrow); voltage bias and thermal noise are supplied by the resistor (right).

Cerous Magnesium Nitrate Temperature Scale.—Cerous magnesium nitrate (CMN) has been gaining widespread use as a medium for obtaining and determining temperatures below 2 K. Since previous studies on this salt have provided a temperature scale of somewhat questionable reliability, a reinvestigation of the temperature scale was undertaken, and a relation between the thermodynamic temperature and the magnetic susceptibility of a single-crystal sphere of CMN was deduced. The results disagree with earlier measurements, but are in reasonable agreement with recent work obtained by a different method.

Improved Thermometer Calibration Service.—An improved calibration service is now available for standard platinum resistance thermometers. Recent improvements in techniques and apparatus permit a wider variety of such standard thermometers to be calibrated more accurately and in a much shorter time than before. The calibrations over most of the range from 10 to 900 K can now be made with errors of less than 0.002 K. The turnaround time for this service has been reduced to six weeks.

Nuclear Quadrupole Resonance Thermometer.—The nuclear quadrupole resonance frequency of ^{35}Cl in KClO_3 in the temperature range 12 to 297 K has been measured. The frequency appears measurable to sufficient accuracy to yield uncertainties of ± 1 mK in the range 50 to 297 K, ± 4 mK at 30 K, and ± 10 mK at 20 K.

Liquid Hydrogen Vapor Pressure-Temperature Scale.—Using germanium resistors as transfer thermometers, vapor pressures of equilibrium liquid hydrogen in the temperature range 13.8 to 20.3 K have been directly related to values of temperature on the Thermodynamic Temperature Scale as realized by the NBS Acoustical Thermometer. Thus a temperature Scale of liquid hydrogen vapor pressure versus temperature has been realized. The results were of particular value to the Comité Consultatif de Thermométrie and aided in the Comité's deliberations which resulted in a new International Practical Temperature Scale. The new scale terminates at 13.8 K; the previous scale terminated at 90 K.

High Temperature Gas Thermometer.—The high temperature gas thermometer at NBS reached operational status. It is a constant volume type, employing a 500 cm³ Pt-20 percent Rh bulb with helium as the thermometric fluid, and is designed to determine corresponding values of temperature on the Kelvin thermodynamic and International Practical Scale every 50 deg between 273 K and the gold point, 1336 K. So far the apparatus has been used at 273 K and 373 K. The measurements were reproducible to within 1 ppm of the absolute temperature (± 0.0004 K at the steam point) for each of the initial pressures of 10, 20 and 30 cm Hg. The stability of the thermometer was demonstrated by repeatability of the results when the thermometer bulb was cycled between 0 and 100 °C over time periods up to 6 days. Exact values of thermodynamic temperature will be computed when measurements of apparatus characteristics are completed.

Improvement in Optical Pyrometer Calibrations.—The accuracy with which high precision automatic optical pyrometers can be calibrated has been improved by a factor of about 3. Typical standard deviation uncertainties now range from about 0.3 deg at 1063 °C to

1.0 deg at 2000 °C and 4 deg at 3500 °C. A major factor in this improvement has been the development of a very stable blackbody which can be operated to about 2500 °C and is used to transfer the International Practical Temperature Scale from the NBS Photoelectric Pyrometer to the test instrument. Another significant contribution in reducing the uncertainties has been the development of a special type composite filter whose transmittance is an insensitive function of temperature. These filters are now being used in high precision commercial pyrometers and make the higher ranges of these instruments significantly more stable.

Copper-Point Blackbody.—The long-term instability of pyrometer lamps used in high precision automatic pyrometers has created a need for a stable inexpensive source of optical radiation for monitoring these lamps. A simple furnace and blackbody, stabilized at the melting temperature of copper, has been developed for this purpose. The source requires no inert gas or vacuum, and utilizes only 225 watts to reach a melt about an hour after turn on. The blackbody can be stabilized at the copper point to within 0.1 °C for 10 min or longer. Using the standard sample copper available from NBS, the radiance temperature of this blackbody source has been determined to be 1083.3 °C with an uncertainty of about 0.1 °C and a reproducibility (standard deviation of a determination) of 0.01 °C.

Electron Density Determination from Spectral Line-Profiles.—The number density of electrons in gases at high temperatures is a temperature sensitive quantity useful in measurements above 10,000 K for many common gases. When the high temperature source contains some hydrogen, the electron density can be obtained by determining the spectral line profile of the H_β line at 4861 Å and by using the theoretical calculations of Griem et al. The accuracy of 10 percent claimed for these calculations has recently been confirmed by measurements on a high current atmospheric pressure arc in argon seeded with hydrogen. Under conditions of thermodynamic equilibrium at atmospheric pressure, the maximum attainable electron density in argon is $2 \times 10^{23} \text{ m}^{-3}$ at 16,700 K. Arcs operated with higher axial temperatures exhibited H_β line profiles which at their off-axis point of maximum broadening agreed with the theoretical profiles for this electron density well within 10 percent uncertainty of the theory.

Electric Current

Absolute Ampere Determination.—The value of the NBS ampere was determined in absolute measure, using the recently modified NBS Pellat-type dynamometer. The ratio of the NBS ampere (derived from the units of emf and resistance as maintained by the NBS reference standards) to the absolute or theoretical ampere (defined in terms of

the mechanical force between current-carrying conductors) was found to be $A_{\text{NBS}}/A = 1.000010 \pm 0.000007$. The stated uncertainty represents the estimated probable error (50% confidence interval) resulting from all known sources, the estimate being based on measurement repeatability. This result, together with other recent absolute determinations of the electrical units will be considered by an International Committee later this year in reassigning the units of emf which are maintained by the various national laboratories.

International Comparison of Capacitance Standards.—Three stable 10 pF fused-silica capacitors constructed in 1964 continue to be circulated to various national laboratories for comparisons of the unit of capacitance. The most recently completed circuit was NBS, NSL (Australia), ETL (Japan), and NBS. Values in the NBS laboratory at the beginning and end of this circuit agreed within a part in 10^7 , indicating that the construction is adequate for international comparisons. The group of standards of this type held at NBS have now been under observation for more than three years and appear to be stable within this same limit.

Interlaboratory Agreement of Electrical Measurements.—Statistical experiment designs and methods for data analysis were provided as part of the Bureau's collaboration with the Air Force on precision electrical measurements. The designs are used in carrying on an interlaboratory comparison of calibrations and for methods of transporting voltage standards.

Interlaboratory Transport of the Unit of Voltage.—The National Bureau of Standards and the U.S. Air Force undertook a joint study of the interlaboratory transfer of the unit of voltage. The two major objectives of the program, both of which were achieved, were to reduce the uncertainty of the assignment of the local unit and to reduce the time required to make the assignment. Using the present method, six to eight weeks are generally required to assign the unit with a stated uncertainty of 1 ppm. The techniques developed in this program reduce the time required to about two to four weeks with an overall uncertainty of approximately 0.5 ppm.

Fundamental Physical Constants

The Faraday.—An analysis was made of differing values of the Faraday—most of which have been determined at NBS. Values obtained by silver deposition, iodide oxidation, oxalate oxidation, the omegatron, and silver dissolution were reviewed. All values were converted to the unified ^{12}C international scale of atomic weights using the international atomic weights of 1967. The value determined

by dissolution of silver in aqueous solutions was shown to be least subject to uncertainties. A value of $96,487.0 \pm 1.6$ coulombs per gram-equivalent was recommended.

Laser Velocity of Light Measurement.—Important progress has been made in the refinement of laser stabilization and control as applied to the measurement of the velocity of light. The method involves measuring the difference in wavelength and frequency of two laser lines very accurately and using these measurements to calculate the velocity of light. Three important steps have been taken: (1) the 51 GHz photobeat between a pair of laser lines at $1.15 \mu\text{m}$ has been measured, (2) the stability of the 30 m interferometer has been studied using a laser which is locked to a stable 30 cm reference cavity, and (3) apparatus has been developed which allows locking an auxiliary laser at a known variable frequency offset from a reference laser.

Mechanical Quantities

Adiabatic Saturation Psychrometer.—NBS has developed an adiabatic saturation psychrometer for measuring the humidity of gases. This instrument can also be used to determine the vapor content of a vapor-gas mixture. The psychrometer behaves in accordance with predictions deduced from thermodynamic considerations, and has considerable potential as a standard for meteorological use. Since it yields a direct measurement of "thermodynamic wet-bulb temperature," it should have application to measurement problems in engineering fields, particularly in air conditioning. In the chemical engineering field, especially in several of the process industries, the instrument can be used to measure the vapor content of various vapor-gas mixtures.

Wide Range Vibration Generator.—Piezoelectric vibration generators (shakers) have been developed which produce motion suitable for the calibration of vibration pickups over a frequency range of 1 to 60 kHz. These shakers incorporate highly elastic, highly damped coupled resonators with overlapping resonant frequencies, and they produce relatively good motion over a wide frequency range. Indications are that these techniques can be extended to frequencies of at least 100 kHz. The measurement of small amplitude, high frequency mechanical vibrations is especially important in the scale model testing of large complex structures such as space vehicles.

Hydrostatic High-Pressure System.—A cylindrical high-pressure liquid container which uses a polyethylene liner to seal the high-pressure liquid has been developed. The container can be pressurized in almost any conventional piston and die press and used to perform PVT and ultrasonic measurements on liquids and solids immersed in

liquids at pressures as high as 45 kbar. Electrical leads have been brought into the high-pressure container in order to determine electrical properties of materials as a function of pressure. Ultrasonic measurements with water pressurized in the liquid container were used to compute the density of water at 22 °C to 12.6 kbar. The estimated uncertainty of these measurements is 0.004 kbar.

Microphone Test Calibrations.—A new method for performing test calibrations on standard condenser microphones has been developed. The method makes use of the reciprocal properties of NBS reference standard microphones. Instead of making a direct comparison of the test microphone against the reference standard using a third microphone as a sound source, the standard is used as a source and the test microphone as a receiver. Only one reading is necessary to measure the response of the test microphone at each frequency rather than two as previously required. Test calibration time is reduced by up to one-half.

Fatigue Crack Detector.—Fatigue tests of high strength aeronautical fasteners loaded in double shear require detection of the fatigue crack in the fastener before catastrophic failure of the fastener occurs. A detector with associated circuits was developed which senses the change in stiffness of the fastener while it is subjected to cyclic loads. The heart of the device is a linear-variable-differential transformer whose output is recorded and which automatically causes termination of the test a few hundred cycles after the crack initiates. In most cases, the resulting crack is too small to be visually detected but its presence is verified using a dye penetrant.

Volume Changes Accompanying the Extension of Rubber.—As part of the effort to determine how to include the effect of compressibility in a general, nonlinear, constitutive equation, the volume changes accompanying extension of peroxide vulcanizates of natural gum rubber have been measured with a dilatometer. These volume changes, of the order of 10^{-4} cm³/g for 100 percent extension, were measured with a precision of ± 2 percent. Measurements of force-extension behavior and compressibilities were made on the same samples. Though a constant compressibility described the results obtained using isotropic pressure, the volume changes accompanying extension were not proportional to the isotropic part of the stress. Thus, the strain energy in extension cannot be separated into a sum of two parts, one due to the shear and one to the dilatation.

Determination of Structural Damping.—Structures such as turbine blades, air frames, and rocket housings require not only optimum strength but also proper damping characteristics. A method to deter-

mine the damping coefficient of structural metals has been developed. Vibration tests of mass-loaded, cantilever-beam specimens were made in air and in vacuum. The method utilized a knowledge of the effects of mass-loading to normalize the experimental data to fit a nondimensional curve of transmissibility—the ratio of free-end to fixed-end beam displacements.

Cryogenic Flow Research.—A program is being developed for an analytical and experimental verification of the performance of existing flowmeters under well defined reproducible conditions. Work is also underway to develop recommended measurement practices for use of these meters in cryogenic systems, and to investigate new commercial metering devices. Implementation of these objectives will provide Government and industry with a centralized research facility for the study of cryogenic flowmetering problems. The program was established under sponsorship of NBS and the Compressed Gas Association. The joint sponsorship by CGA provides international support for the program as this trade organization represents six of the largest cryogenic producers in the United States as well as cryogenic producers in England, France, and Germany.

Swirling Flow Study.—The presence of swirling flow can at times have significant influence upon the accuracy of flowmeters, including turbine and differential-pressure types. However, the behavior of swirling flow fields is not well known. Cooperative studies with the University of Maryland Department of Mechanical Engineering on turbulent, incompressible swirling fields in long, cylindrical, unobstructed pipes show that the angular momentum flux decays exponentially along the pipe at a rate of about 3 percent per pipe diameter. Also, the rate of decay increases slightly as the Reynolds number decreases in the range from 200,000 to 12,500. Information from these studies will aid in an improved description of swirling fields in which the performance of flowmeters can be evaluated.

Electrical Quantities—DC and Low Frequency

Improved 10,000- Ω Resistance Calibrations.—For half a century standard resistors of the “NBS Type” have been widely used in electrical standardizing laboratories in a range of nominal values extending from 1 Ω to 1 M Ω . Demands for better standards at kilohm resistance levels having stability comparable to the 1- Ω level are being met with improved 10,000- Ω standards recently developed by several manufacturers. NBS has developed methods and constructed apparatus for calibrating these improved standards with a total measurement uncertainty of 1 ppm. This uncertainty applies to the calibrated value (given to the nearest 0.001 Ω) in terms of the legal unit of resistance maintained with 1- Ω standards. To obtain this increased accuracy,

better methods for scaling resistance values have been developed utilizing so-called "Hamon Devices" for multiplying resistance values 100-fold from 1 to 100 to 10,000 Ω . In practice, the accuracy of these devices approaches the theoretical limit (ratio uncertainty a few parts in 10^9).

Cryogenic Parametric Amplifier-Detector.—A null detector for audio-frequency capacitance bridges has been developed. The device, which utilizes solid-state varactor diodes, permits an increase in measurement resolution up to a factor of five over conventional units. Operated at room temperature using a signal frequency of 10^4 rad/s, a minimum noise figure of 0.01 dB was obtained; at liquid nitrogen temperatures, noise figures below 0.001 dB (referred to a room temperature source) were measured.

Standard Cells.—Seventeen Vosburgh-type, saturated Weston standard cells were constructed and their emf's measured from 5 to 40 °C. This type of cell (Bi added to Cd-Hg anode and double salt $\text{CdSO}_4 \cdot \text{Na}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ added at both electrodes) has an emf (1.018690 V) at 25 °C which is almost the same as the conventional, saturated Weston cell (1.018393 V), but it has a lower emf-temperature coefficient: $+13.5 \mu\text{V}/^\circ\text{C}$ compared with $-49 \mu\text{V}/^\circ\text{C}$ for the conventional type. This cell is expected to have a long-range emf stability.

Voltage-Ratio Measurements.—The "boot strapping" technique for evaluation of inductive voltage dividers has been further improved in accuracy and expanded in frequency coverage. It is now estimated that an accuracy of 2 parts in 10^7 is obtainable for 5 and 10 kHz calibrations, and a few parts in 10^8 for 400 and 1000 Hz calibrations. Since it is a self-calibration method, the technique does not rely on any national standards, so it may be used by any laboratory with equal validity.

High-Voltage Pulse Measurements.—A Kerr electro-optical system for measurement of high-voltage pulses has been developed and calibrated. This system permits time-resolved measurements of pulses with peak amplitudes as high as 100kV. Simultaneous pulse divider and Kerr system measurements of pulses as high as 100 kV have indicated that the Kerr system is accurate at least to the same degree as the pulse divider (to within 1 percent). With further refinements, it is anticipated that the system will be useful as a standard for calibration of pulse dividers and that it will permit measurement of transient voltages considerably in excess of 100 kV.

Stable 10 kV Power Supply.—A stabilized adjustable high-voltage supply with a direct current capacity of 10 mA has been built for use

in an absolute volt determination. Its drift rate, measured over time intervals greater than a minute and extending to more than 10 hr, is less than 1 part in 10^7 /hr. For shorter times (1 s to 1 min) the peak-to-peak deviations from the mean are less than ± 2 parts in 10^7 at 10 kV. Facilities are included to permit accurate measurement of the output voltage of the supply against standard cells.

Stark-Effect Voltmeter.—Work has begun on the development of a voltage standard at the 10 kV level based on the Stark effect of molecular radio frequency spectra. An ultimate precision of 1 part in 10^6 is being sought; absolute accuracy depends upon the accuracy to which the electric dipole moment of the working molecule is known. Initial experiments were performed with a beam maser spectrometer. Following successful performance tests, the maser was modified for Stark effect measurements on the UHF spectra of hydrogen cyanide (HCN), formaldehyde (H_2CO) and deuterated water (HDO).

Electrical Quantities—Radio Frequency

Radio Standards from 30 KHz up to and including microwave, millimeter wave, and laser frequencies are provided by the Radio Standards Physics Division, the Radio Standards Engineering Division, and the Time and Frequency Division of NBS, located at Boulder, Colo. These groups conduct basic and applied research leading to standards for important radio quantities (such as power, pulsed quantities, attenuation, and noise) and to standards of frequency and time. They also perform calibration and broadcast services which disseminate standards to industrial and Government laboratories.

High Frequency Region

3 MHz Noise Calibration.—A new calibration service for noise measurements at 3 MHz was completed and announced. The service is for thermal and shot noise generators having a noise temperature in the range from 75 K to 30,000 K. The service is capable of a maximum uncertainty in the measured value of noise temperature of approximately 1 percent. This is the first announced service for noise in the high frequency region and in coaxial systems. It uses a new technique of measurement for which a patent has been issued. Noise is an important quantity in many areas of the radio electronics field. The sensitivity of a receiver is ultimately limited by the noise which it generates internally and receiver sensitivity, in turn, directly influences how soon a missile is detected or how well a deep space probe functions.

Improvement in Coaxial Power Measurements.—A new rf-dc substitution calorimeter with automatically controlled dc reference power

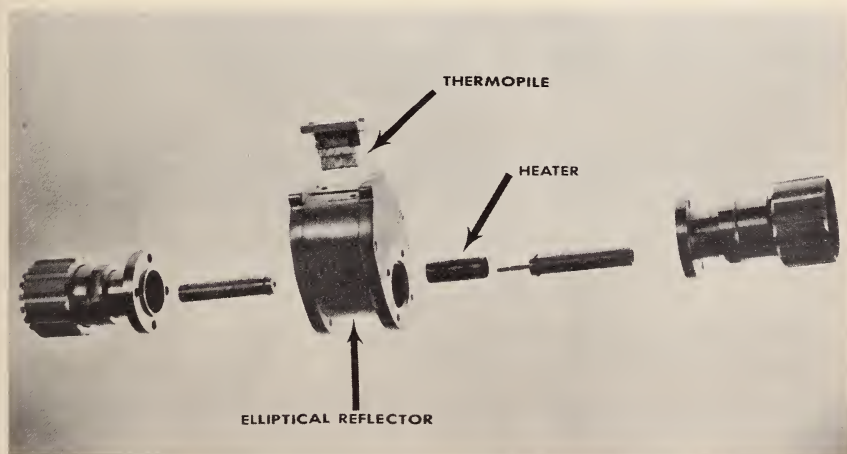
has been developed that allows improved accuracies of measurement in the lower power ranges. The new standard extends the range of the existing coaxial reference standards in the frequency range below 1 GHz from 50 mW to 10 mW making possible the direct calibration of 10 mW bolometer mounts. The measurement range of this new standard is from 10 mW to 1 W at frequencies up to 4 GHz and beyond. Maximum measurement uncertainty is 0.35 percent. This standard was designed so that it can be connected to digital equipment for direct automatic recording of measurement data.

Superconducting Transmission Lines.—A miniature, 80-foot coaxial line (Nb inner conductor 0.01 in O.D., Pb outer conductor 0.034 in I.D., polytetrafluoroethylene dielectric) has been analyzed in terms of the Gorter-Casimir two-fluid superconductivity model considering the anomalous and classical cases (without relaxation effects) for the normal component. The line attenuation 10^3 to 10^{16} Hz and the step response from 1 to 100 ps have been calculated for the temperature range 2.3 K to 10 K. This analysis, and its comparison with the experimental results from other independent investigators have established the bounds between which the responses of physically realized superconductive lines will lie. Additionally, the results present a method of identifying the purity of the conductors in a coaxial line of this type. The study of coaxial transmission lines at superconducting temperatures is important because of their ability to transmit pulses over long distances with minimum degradation of pulse shape and also because of their potential for transmitting large amounts of power with a minimum of loss.

Automation of High-Frequency Power Measurements.—The high-frequency, coaxial bolometer calibration console was fully automated with the addition of a small on-line computer. This enables the collecting, compiling, and computing of the output data from the console to yield the final calibration result. The calibration time has been reduced by one-half, as has imprecision of the measurement. The human error involved in compiling and computing the output data has been eliminated.

Peak Pulse Power Calibrations.—Calibrations of coaxial peak pulse power meters in the frequency range 300 to 500 MHz have been established in the range of 1 mW to 2.5 kW peak power. The pulse width can be calibrated in the range from 2 to 10 μ s with a pulse repetition rate of 100 to 1600 pulses/s, and a maximum duty factor of .0033. Peak pulse power measurements are important in the determination of output power from such devices as radar systems and pulse-modulated information transmission systems.

Portable Current Standard.—A small reference standard of radio frequency current has been developed which can serve as a high accuracy, interlaboratory standard. The ammeter is unique and has no precedence in principle of operation. It consists of a cylindrical, ellipsoidal silver reflector of infrared energy with a cylindrical thin-film quartz heater along one focus axis and a heat sensing thermopile along the other. The heater forms an extension of the center conductor of a coaxial line, and the ellipsoidal reflector forms an extension of the outer conductor. The ammeter has three times the current range of previous devices, up to 10 times larger output for a given sensed current and unequaled broad-band frequency range. The prototype covers the range from 0.5 to 5 A. The dc output corresponding to 0.5 A is approximately 1 mV and that corresponding to 5 A is approximately 100 mV for currents in the frequency range of dc to 1 GHz. The ammeter is the first to use coaxial transmission line design which makes it compatible with 50- Ω coaxial systems without introducing additional uncertainties. The ammeter may also be useful as a wattmeter or voltmeter in special circumstances.



NBS-developed reference standard for interlaboratory comparison of radio frequency current.

HF Electrodynamometer Accuracies Determined.—The high-frequency short-circuit ring electrodynamicometer developed at the NBS Boulder laboratories has been more accurately analyzed as a result of a new technique for obtaining the ratio of the torque on the ring to the current on the transmission line. Application of this technique to the electrodynamicometer has shown it to be capable of measuring currents

over the frequency range 1 MHz to 1 GHz within the following limits of uncertainty: at 1 A the uncertainty is within ± 0.06 percent, and up to 100 A it is within ± 0.5 percent.

A New Standard of Voltage, Current, and Power.—A new standard of voltage, current, and power, referred to as the “Bolovac,” was developed for lumped-constant, coaxial, and other circuits operating in the TEM mode. Its approximate ranges are 1 MHz to 20 GHz in frequency, 0.05 to 10 V, 5 mA to 10 A and corresponding power levels in 50- Ω systems. A thin film conducting element located in the transverse plane of the TEM mode is the principal sensing element of the device.

Broad Band Inductive Voltage Divider.—A new technique has been devised for making broad band inductive voltage dividers of the 2 to 1 ratio type. This type of divider, which has a ratio of input voltage to output voltage of 2^n for cascaded transformers, has had until now a very high accuracy only at a fixed frequency. The new technique compensates for the voltage drop across the leakage impedance by placing a small impedance between each transformer and ground. By making this small impedance from the same type of cable used to construct the transformers, it can be made to compensate for the leakage impedance over all frequencies for which the transformers are usable; i.e., 1 kHz to 1 MHz. The work shows that it is now possible to build a cascaded inductive voltage divider of this type with uncertainties in the order of 10^{-5} over the entire frequency range.

Radio Frequency Standards Comparator.—An rf standards comparator, capable of providing standards of radio frequency at the user level has been developed. The comparator consists of a voltmeter, ammeter, power meter, and an impedance bridge combined in a compatible, 50- Ω , coaxial system in which the measured values of any one instrument can be compared to results obtained from the other three. Intercomparison of the electrical quantities of power, impedance, voltage, and current is possible over the frequency range from 30 MHz to 1 GHz with a precision of about 0.1 percent. These quantities are related mathematically, so that if any two are known the remaining two may be computed; if three are known, a redundancy exists which permits a self-checking system. If all four quantities are known, a double redundancy exists which adds an extra degree of confidence to the standards.

Microwave Region

Low Temperature Noise Standard.—An X-band (10 GHz) thermal noise source has been developed. It consists of a waveguide termination in a cryogenic environment along with temperature and pressure monitors and controls. The effective noise output at the room temperature

flange can be set to the boiling point of helium (4.2 K) with an accuracy of ± 0.03 K. The noise source can also be used with liquid nitrogen as the coolant for which the effective noise output temperature is then approximately 77 K with an accuracy of ± 0.16 K. Temperature stability is 0.002 K for helium, and 0.02 K for nitrogen. At present, the useful accuracy of the standard is approximately 0.1 K; the primary source of uncertainty is the insertion loss (approximately 0.001 dB) of mating room temperature flanges.

Microwave Noise Measurement.—A technique has been developed for the comparison of low temperature microwave noise sources independent of their reflection coefficients. At a noise-source temperature of approximately 77 K, noise-power output of sources with reflection coefficients as high as 0.5 can be compared within uncertainty limits of ± 0.1 percent relative to a noise standard. This technique can be applied at any microwave frequency range for which three-port circulators are available, and at higher noise-source temperatures.

Microwave High Power Measurements.—A technique has been developed for calibrating directional coupler bolometer units used for high-power microwave measurements. The technique virtually eliminates mismatch errors, reducing the uncertainty in the calibration of high coupling ratios from 15 to 1.5 percent. Because it employs existing power measuring equipment and standards, it should lead to wide adoption and use by industry.

International Attenuator and Impedance Comparisons.—Attenuation measurements were compared with those of Sweden. The comparison was made on a variable attenuator in WR 90 waveguide at a frequency of 9.0 GHz. Agreement was within 0.03 dB for most values measured. The first phase of an intercomparison of impedance standards with Italy has also been completed. The reflection coefficient magnitude of a WR 90 fixed waveguide reflector was measured at 9.0, 9.8, and 11.2 GHz.

Electromagnetic Field Standards

Near to Far Field Antenna Pattern Calculations.—Work on the establishment of methods to calculate far field antenna patterns and antenna gain from near zone data is rapidly approaching successful completion. The results of additional experimental data indicate a strong probability of success. Computer programs have been developed which allow the far field to be represented in a graphic projection of three dimensions. Successful completion of this work will result in a significant saving in the cost of determining the far field of large aperture antennas.

Measurement Capability in Field-Strength Calibrations.—The capability for measuring field-strength and other precision receivers from 1 to 10 GHz has been completed and announced. This new service includes calibrating such receivers for use as two terminal rf voltmeters, calibration of signal attenuators, and calibration of overall receiver linearity. The rf voltmeter calibration is performed in terms of coaxial power standards, and attenuation and overall linearity measurements are accomplished using the rf substitution method. With this new measurement capability, the frequency range of the field-strength receiver calibration service is 30 Hz to 10 GHz.

New Calibration Services

The following is a complete list of services completed and announced during FY-68:

- Reflection Coefficient Magnitude in WR 284 and WR 42 Waveguide,
- Power Measurements of Bolometers and Bolometer-Coupler Units in WR 284 Waveguide,
- Reflection Coefficient Magnitude for Non-Reflecting Waveguide Ports in Waveguide Sizes WR 284, 187, 137, 112, 90, 62, and 42,
- Phase Shift Measurements of Variable Phase Shifters in WR 90 Waveguide,
- Power Measurements of Bolometer Units in WR 28 Waveguide,
- Improved accuracy of Coaxial Impedance Standards in the range 1-8 GHz,

and the following, which have been discussed in detail above:

- Peak Pulse Power,
- 3 MHz Noise,
- Extended Frequency Range for Calibration of Field Strength receivers.

Thermal Quantities

Thermal Expansion Measurements.—Equipment is now available to measure linear thermal expansion of solids from 10 K to 1,900 K. Materials under consideration for standard reference samples (e.g., copper, fused silica, and sapphire) have been measured over this extended temperature range. In the range from 10 to 1,200 K, a dual interferometric apparatus was developed to measure the samples to an accuracy of 1 $\mu\text{m/m}$. From 1,100 to 1,900 K a comparative technique was developed with an accuracy of 30 $\mu\text{m/m}$, the most accurate available in this temperature range.

Photometric and Radiometric Quantities

Continuous Wave Laser Power Meter.—A photoelectric meter was constructed for measuring the power output of CW lasers. The calibration factor of the meter was determined by four radiometric techniques: (1) a filter radiometer calibrated with an NBS lamp standard of spectral irradiance, (2) a prism spectroradiometer similarly calibrated, (3) a radiation thermopile calibrated with an NBS lamp standard of total irradiance, and (4) an absolute radiometer calibrated by electrical heating. The resulting calibration factors of the meter were consistent to ± 2 percent. Although this work involved a particular 80-mW He-Ne laser operated at a 6328 Å, the techniques are applicable to a much larger class of calibration problems. In addition, the results obtained indicate the consistency of the calibrations of NBS standards of spectral and total irradiance with each other and with the electrical unit of power, the watt.

Laser Energy Power Measurements.—A calorimeter containing aqueous CuSO_4 and designed to measure pulsed laser energies to 10 J/cm² at peak power to 200 MW/cm² has been developed. In the energy range 1 to 100 J, a comparison with other energy measuring devices indicates an accuracy of ± 2 percent. Intercomparison of calibrated calorimeters suggests a precision of 0.7 percent. In addition, the average power of CW lasers can be measured in a similar manner with an indicated accuracy of ± 4 percent.

Infrared Laser Power Measurements.—A new type of calorimeter utilizing an absorbing gas as the heat transfer medium has been designed and built. The device will be tested on a CW 50 W CO_2 -N₂-He commercial laser. Initial calculations indicate that 100 and 1000 W sources can be measured without major modification.

Production of Side-Bands on Visible Light.—Visible light from a helium-neon laser has been modulated at a microwave frequency of 10 GHz and the two resulting side-bands have been observed. The effect was produced by placing a crystal of KDP within the laser cavity and also in a microwave cavity excited by a 10 GHz microwave oscillator. The output light from the laser was observed to contain not only the normal 6328 Å red light, but also two sidebands, one at a wavelength corresponding to the frequency of the red light plus the microwave frequency, the other at the difference frequency. An output of 10^{12} photons per second was observed in this side-bands.

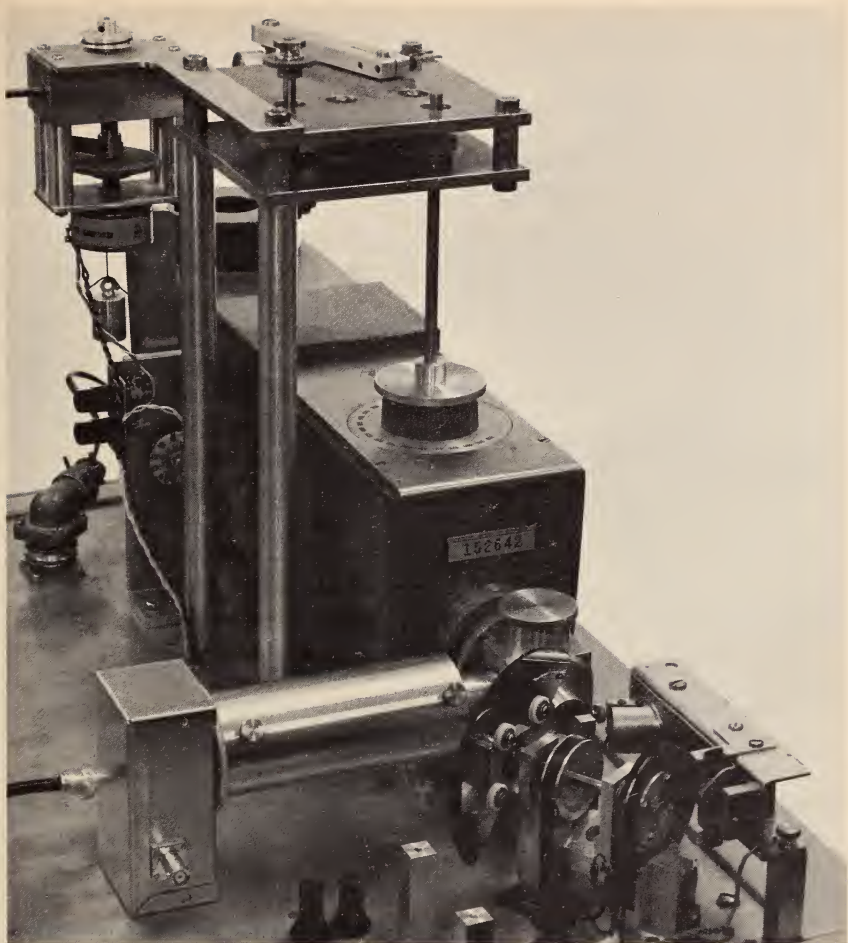
New Image Converter.—A new way of seeing and photographing objects by invisible infrared and ultraviolet rays has been demonstrated. When energy from an object was focused on a special plate, a

color change occurred at the image, making the image visible. The plate, which may be used repeatedly, was prepared by laser exposure of a high-resolution photographic emulsion in contact with mercury. The standing wave pattern of interference of the incident and reflected beams was recorded in the emulsion which was then developed. The color of such a plate is so sensitive to relative humidity that the slight change in local humidity due to an infrared image is readily seen. The new principle has been demonstrated only under laboratory conditions but offers promise of very simple and economical devices for a number of infrared and ultraviolet applications.

Standard Detectors for the Vacuum Ultraviolet.—An investigation into the day-to-day reproducibility of the photoelectron yield of materials in the vacuum ultraviolet has been initiated. Given a material with adequate stability, a calibrated sample could serve as a transfer standard detector for use in this region. It has been discovered that the yield of materials is extremely sensitive to the degree of surface contamination present. Thus far, studies have eliminated some materials from consideration because of excessive sensitivity to ambient temperature or to storage history. Other materials are showing promise for use either at ambient temperature or at temperatures sufficiently high to preclude the influence of contamination from the vacuum system housing the sample.

Photodetector Calibration Service Expanded.—Calibration of photodetector response can now be performed over the spectral region 0.35 to 1.8 μm with a resolution of approximately 3.5 nanometers in the visible region and 7 nm in the near infrared. The relative spectral response of a photodetector can be measured for unmodulated (dc) light, or for light modulated (ac) at ten fixed frequencies from 1 Hz to 1 kHz. The absolute spectral response can be measured at certain fixed wavelengths. Uncertainties in spectral response are less than ± 3 percent of peak response for relative measurements and less than ± 5 percent of peak response for absolute measurements.

Infrared Spectral Radiance Calibrations.—A blackbody and spectroradiometer have been developed which are capable of determining the spectral radiance of 500 to 1300 K test blackbodies between about 1 and 4 μm with an estimated uncertainty of 0.1 percent or the equivalent of 0.4 K, whichever is greater. The reference blackbody consists of a 1.5 mm thick graphite cavity embedded in copper, and the spectroradiometer consists of an f/4 prism monochromator, a liquid nitrogen-cooled PbS detector, a "lock-in" amplifier, and means for precisely orienting and translating the blackbody sources. The thermometer presently being used to determine the blackbody temperature is a primary-calibrated platinum versus platinum-10 percent rhodium



Photodetector response is calibrated by this machine which measures detector output under monochromatic light swept across part of the electromagnetic spectrum.

thermocouple. This work has been largely motivated by infrared requirements in the Department of Defense, and NBS is working with the Newark Air Force Station to establish the capability of accurate infrared radiance calibrations.

Evaluation of Lenses for Aerial Color Photography.—Aerial color photography is finding increasing application in agricultural and geological surveys, mapping, and reconnaissance. To evaluate lenses for such uses, the camera calibration laboratory at NBS has been equipped to expose color materials to test patterns illuminated by light of the three primary colors employed in color photography. The

images can now be evaluated separately. Three normal color films and one false color film have been used in the evaluation of resolving power, chromatic aberration, and field curvature of seven lenses in wide use or of advanced design.

Standard Nomenclature for Radiation, Light, and Color.—Members of the NBS staff directed efforts that led to the publication of USA Standard Nomenclature and Definitions for Illuminating Engineering. Although this standard (Z7.1-1967) was developed for illuminating engineering, it is of even greater interest in the fields of photometry, colorimetry, and radiometry because it contains terms, definitions, and symbols in these fields that are entirely consistent with those adopted by committees of the International Commission on Illumination, the International Organization for Standardization, the International Electrotechnical Commission, and the SUN Commission of the International Union of Pure and Applied Physics. This is the first standard published in this country, and possibly in the world, that contains up-to-date terminology in the fields of photometry, colorimetry, and radiometry of such widespread acceptance that it can reasonably be expected to remain authoritative for many years.

Survey on Colorimetry.—A review of the field of colorimetry has been completed and made available. This review includes, in addition to subject matter retained from earlier documents, the most important recent developments in colorimetry: the large-field supplementary observer system, the new sources that represent daylight, and the treatment of colorimetry as a measurement science with associated uncertainties.

Universal Color Language Applications.—The ISCC-NBS method of designating colors, now known as the Universal Color Language, has become the link between simple color designations (red, blue, yellow, green) and the numerical specifications of color. It consists of six levels of precision to supply the appropriate degree of accuracy in color designation needed by science and industry. NBS utilizes this system to help those with color problems. Two examples of its use this year are the color designation system of the Manual of Photogrammetry of the American Society of Photogrammetry, and the 12-color code for highway signs in the Manual on Uniform Traffic Control Devices for Streets and Highways. Special attention was directed to the needs of colorblind drivers (about 3 percent of all drivers) during the development of this new code.

Flattery Index Proposed for Lamps.—People and materials look worse than they really are under some lights, and better than they really are under others. There is an international method for measur-

ing the degree to which a lamp succeeds in rendering object colors truthfully. This method has now been modified to indicate how well a lamp succeeds in flattering them. This "flattery index" is under study by the Committee on Color Rendition, Illuminating Engineering Society, for validification and refinement before being recommended for practical use.

Theory of Chromatic Adaptation.—Two objects which look like they have the same color to the eye (i.e., a matched pair) do not necessarily reflect light in the same wavelength pattern. If one stares at a colored light of moderate brightness and then looks back at the matched pair of objects, the match remains but the perceived color changes. When this colored light is very bright, not only does the perceived color change, but also the match is lost. In an attempt to understand these facts about color matching in terms of changes occurring in the visual pigments of the eye, a mathematical study of the bleaching of pigments in solutions was undertaken. It was found that if the pigments absorb between 55 and 85 percent of the light to which they are most sensitive, it is possible to account for both the match breakdown at high brightness and the match preservation at moderate brightness in terms of pigment bleaching alone.

PHYSICAL PROPERTIES

Approximately half of the activity within the Institute for Basic Standards is related to the measurement of physical properties of well-defined substances. The rapid growth of the physical sciences has provided scientists with many new techniques for measuring "old" physical properties and has made accessible many new ones. With new techniques it has been possible to increase the precision of measurement, provide the scientific community with a greater range of reference data, and characterize certain processes more fully. Examples of these activities, drawn from current work, are given below.

Atomic and Molecular Properties

Absolute Electron Scattering Cross Sections.—Further development of electron scattering apparatus has now made possible the absolute measurement of differential cross sections to an accuracy of about 5 percent. Absolute measurements in helium for 2^1P excitation and for elastic scattering have now been completed. Together with very precise (about 1 percent) measurements of angular distributions, the absolute measurements show that contrary to previous assumptions, the Born approximation is not valid at 400 eV, either in absolute magnitude or in the shape of the angular distribution.

Radio Plasma.—The abnormal negative glow plasma in helium has been adapted for a more thorough investigation of the microwave cavity technique used to measure electron density in plasmas. The use of this plasma, in combination with a low frequency microwave cavity, has generated an acceptable situation for experimental and theoretical investigations of the influence of longitudinal modes on the microwave measurements. A double perturbation technique allows for evaluation of the microwave field within the plasma and for an extension of the cavity measurements beyond the limits of the simple perturbation method. Present data indicate that use of the microwave technique as interpreted by the simple perturbation theory is more limited than accepted in the literature due to the presence of excited longitudinal modes not accounted for by the theory. The double perturbation technique has in some situations extended the limits for the measurable electron density by one to two orders of magnitude.

Absorption Spectra in Rare Gases.—Effort has been directed at the analysis of absorption profiles of resonances in the photoionization continuum; analysis of the energy position and profiles of the resonance structure for neutral argon has been completed. Work has been facilitated by digitizing the transmission data and punching it onto paper tape suitable for computer processing, thus eliminating the tedious job of hand data reduction. A least squares computerized parameter fitting routine has been devised to analyze the data, taking into account the smearing effect that the monochromator slits have on the transmission.

Regularities Among Atomic Oscillator Strengths.—During the course of a critical compilation of atomic oscillator strengths from data in the literature, many regularities among these data were detected and systematically investigated. Three principal regularities have been found: (a) systematic dependency of oscillator strengths on the nuclear charge for a given transition within an isoelectronic sequence, (b) systematic variation of oscillator strengths in spectral series with oscillator strengths usually rapidly decreasing with increasing quantum number, and (c) approximately constant oscillator strengths for certain analogous lines in homologous atoms. These regularities have become a very useful and important tool for analyzing the reliability of existing data and have made possible the determination of many additional oscillator strengths by simple interpolation techniques.

Theoretical Calculations of Atomic Oscillator Strengths.—Extensive calculations of atomic oscillator strengths have been made using the method of configuration interaction to obtain the atomic wavefunctions. Such refined calculations have become necessary since recent

experiments show that configuration interaction effects may drastically change the oscillator strengths of certain transitions. As many as 50 configurations are used to express the electron correlation, and f -values with an accuracy generally in the 25 percent range have been computed for C I, C II, Al I, Si II and the magnesium and boron isoelectronic sequences. Some more traditional configuration interaction calculations have also been made to study the absorption oscillator strength distribution in perturbed Rydberg series. These latter calculations predict, for aluminum, the existence of an autoionizing state which takes up most of the oscillator strength.

Calculated Atomic Transition Probabilities.—The transition array of atomic oscillator strengths has been calculated for Ti II using methods which are an extension of Racah's method for energy level calculations. For the first time it was possible to include configuration interaction in these calculations, and it was found that configuration interaction strongly influences the energy level structure and the oscillator strengths for many of the atomic transitions. For a group of transitions, the gf -values which were calculated are within 10 percent of the experimentally observed values, and in general within 30 percent of the observed values of King and Wobig. The calculations have pointed out that some of the experimental values were obtained under optically thick conditions.

Conversion of Translational Energy.—One of the most important but least studied processes which occur in hot gases is the conversion of translational energy of participating atoms and molecules to internal energy. This process is important in determining chemical reaction rates and rates of collisional processes in stellar atmospheres and laboratory plasmas. A measurement technique has been developed involving resonance fluorescence which is applicable for certain simple cases, and which may provide important insight into the general significance of the collisional transfer of translational energy to internal atomic energy.

"Red-Shifted" Spectral Lines.—A "red-shifted" spectral line is usually interpreted as an indication that the emitter and the observer are receding from each other. It has now been shown that red-shifted lines can be formed by an emitting gas that is expanding toward the observer with a positive velocity gradient. Although the resulting apparent shift is too small to be of cosmological interest, this mechanism does appear to explain red-shifted lines seen in certain nebulae.

Atomic Spectra.—Accurate measurements in the vacuum ultra-violet region have resulted in improved wavelength and energy level data for the spectra Cl I, Cl II, S I, and Mg III. For Cl I more than

1100 lines were determined to $\pm 0.001 \text{ \AA}$, an accuracy sufficient for reference standard wavelengths for most work in the region below 2000 \AA . Knowledge of the atomic energy level structure has been significantly improved for the complex spectra Tc I, Ce I, Pr III, Pm I, Tm I, Tm II, and Th I. For several of these atoms the new results include the energies of some of the lowest and most important electronic configurations. A large plane grating spectograph, with a resolving power of 500,000, has recently been completed and will be used to observe complex spectra.

Photodetachment of Negative Ions.—The photodetachment of H_3O_2^- ($\text{H}_2\text{O} \cdot \text{OH}^-$) has been studied in detail because of its possible importance in the control of electron density in the upper atmosphere. The initial results lend increased support to the speculation that water may play a significant role in the physics and chemistry of the upper atmosphere.

Molecular Gas Laser Studies.—Studies have continued on the fundamental processes involved in molecular gas lasers. More HCN laser transitions have definitely been identified and all of the known DCN laser transitions have now been explained. Energy transfer mechanisms as related to laser systems are also under study. Fluorescence lifetime measurements were made on the 001 level (the upper laser level) of N_2O in order to determine the rate of collisional deactivation of that level. Infrared fluorescence measurements were also made on several other molecules. Some double resonance measurements were made by using a 50 W CO_2 laser as a pumping source and observing the effect on rotational levels monitored by means of a microwave spectrometer.

New Method for Sputtering by Low-Energy Ions.—Ultraviolet absorption spectroscopy together with low temperature matrix isolation has been used to study the energy levels of selected metal atoms and their oxides. In this connection a low-energy sputtering process was devised for the production of metal atom samples. The technique involves the production of low-energy rare gas ions in a microwave electrodeless discharge followed by the bombardment of a metal sample by these ions. The process permits controlled production of metal samples which may be difficult to produce by other methods in a low temperature environment.

Crossed Beam Study of Photodissociation Cross Section of H_2^+ .—Crossed beam techniques have been used to study the photodissociation cross section of H_2^+ from 2537 \AA to $13,600 \text{ \AA}$. The method provides a new tool for studying energy partition in dissociative collisions of electrons with more complicated molecules, and gives fundamental

knowledge of the simplest molecule, H_2^+ , important in controlled thermonuclear research, laboratory plasmas, and astrophysics. Calculations have also been made, and close agreement is found between theory and experiment. Disparities can be attributed to differences between actual and theoretical populations of the vibrational levels of H_2^+ .

Electron Impact Spectrometer for Air Pollution Studies.—Recent improvements in electron impact spectrometers at NBS have made it possible to implement the suggestion that the characteristic energy losses of electrons upon scattering be used as an analytical method. Using the improved apparatus, carbon monoxide in air was detected at concentrations of less than 100 ppm. This spectrometer is competitive in sensitivity with the best currently used instruments and very considerable improvements in sensitivity and compactness are possible.

Spectrum of the ClO Free Radical.—Using a new spectrometer built for the study of free radicals, the microwave spectrum of the ClO radical has been observed for the first time. Rotational transitions of the $^2\Pi_{3/2}$ and $^2\Pi_{1/2}$ electronic states of ^{35}ClO and ^{37}ClO were measured. These studies gave new information on the Λ -doubling parameter and the hyperfine constants, and an improvement by a factor of ten for the values of the bond distance and dipole moment for the states of the radicals.

Vibrational and Rotational Spectra of Simple Molecules.—Infrared and microwave spectra have been studied in a number of molecules which have features of special interest. New measurements have been made of certain unusual transitions in the microwave spectrum of HCN, OCS, CH_3CN , and HCCCN . The gas phase spectra of CH_3Cl and CH_3I have been studied in detail to determine the effect of certain vibrational and rotational interactions. These same transitions have been studied in matrix isolated solid spectra to determine how the interactions differ in the solid phase. New infrared measurements have been made on gaseous HCN to improve knowledge of the energy levels involved in laser action in the HCN laser.

Molecular Energy Levels of Carbon Monoxide.—A detailed analysis of the vacuum ultraviolet absorption spectrum of carbon monoxide has been completed. The so-called Fourth Positive System is the most prominent feature of the spectrum in the region from 1100 to 1600 Å. It has particular significance because of the recent observation of this band system in the solar spectrum. In addition to providing improved values for the energy levels and molecular constants of the states of carbon monoxide involved in this transition, the analysis established the existence of a maximum in the potential energy func-



Electron impact spectrometer for the study of the characteristic energy losses of electrons in gases. This device is now being investigated for use in air pollution studies.

tions of the first excited electronic state of CO. Knowledge of the magnitude of this potential maximum is of interest because of the great importance of the value of the dissociation energy of CO in thermochemical studies.

Alkali Molecular Spectra.—Several lines of the argon-ion laser excite definite vibrational-rotational levels in an upper electronic state of Na_2 , Rb_2 and NaK , causing a relatively simple fluorescence spectrum. Using a Fabry-Perot interferometer crossed with a spectrograph, this spectrum was investigated and molecular constants, potential curves and Franck-Condon-factors were determined. This method

allows a high accuracy because single lines can be resolved. Since the high laser intensity creates a high population in the upper molecular states, one can measure the lifetime and angular-momentum coupling condition of these states with level-crossing techniques.

Solid State Properties

Electron Paramagnetic Resonance.—A technique was developed for the measurement of fast paramagnetic relaxation times which employs the Optical Faraday Rotation. This technique was used to measure the relaxation time of paramagnetic Eu^{2+} in $\text{CaF}_2:\text{Eu}^{2+}$ at cryogenic temperatures.

Theoretical Magnetism.—It was shown that any theory of magnetism based on a one-particle distribution function, such as the Weiss molecular field theory, is inconsistent if it assumes no correlation between spins. The consistency condition, a direct consequence of equilibrium fluctuations, was invoked to provide a measure of two-spin correlations, thereby implying a two-particle distribution function compatible with the one-particle Weiss distribution. The resulting theory is exact at high temperatures for evaluating such inherently two-particle quantities as heat capacity.

Various effective field techniques for the study of magnetism, appropriate for the Ising interaction, were examined with an infinite chain used as the basic cluster. These theories were found to offer no substantial improvement over the analogous theories using very small clusters.

Nuclear Magnetic Resonance.—A new parametric nuclear spin saturation technique has been developed using ^{27}Al nuclei in ruby. With the proper orientation of the c -axis with respect to the external magnetic field and rf magnetic field, magnetic $\Delta m = \pm 2$ nuclear spin transitions were observed. The theoretical calculation of this effect gave excellent agreement with experiment, and indicated that $\Delta m = \pm 3$ and ± 4 magnetic transitions should be observable.

Field-Electron Studies of Adsorbed Atoms.—When atoms are adsorbed on a metal surface, the highest electron energy level of the atom is shifted upward and broadened into a band which may be several electron volts wide. Recently, it was predicted that the distribution in energy of electrons field-emitted from a metal through single atoms on the metal surface would reveal information about the shift and broadening of the atomic energy level. Experiments conducted with single strontium atoms on an oxygen-coated tungsten surface verified the theoretical prediction and established the feasibility of obtaining information about the interaction of atoms and surfaces with this technique.

Electronic Characterization of High-Purity Metals.—A strong correlation between low temperature electrical resistance and chemical purity has been shown for many metals. To exploit this fact an eddy-current apparatus has been developed and refined to allow rapid determination of the residual resistance ratio, which is the ratio of the electrical resistance at the ice point to the resistance at liquid helium temperatures. This contactless method works with bulk specimens and avoids both thin wires and soldered contacts. The method is especially useful for production control or inhomogeneity testing. A large number of metals have been tested for the NBS Standard Reference Material program, including Ag, Al, Au, Cd, Cu, Mo, Pt, Sn, W, Zn, and several noble metal alloys.

Characteristics of Slush Hydrogen.—Activities in the physical characterization of liquid-solid mixtures of hydrogen (slush hydrogen) have continued. A densitometer has been constructed which provides a traceable calibration for slush density instruments used as secondary or transfer standards. At present, the most successful density measuring instruments utilize nuclear radiation attenuation techniques. A parametric study of the performance of a centrifugal pump operating with slush hydrogen was made. It was found that both fresh and aged hydrogen mixtures could be successfully pumped and that performance and cavitation characteristics are the same when the difference in fluid density is considered.

Standard Reference Thermocouple Tables.—Printed and punched card tables resulting from NBS research and analysis are available in 1 K intervals from 4 to 280 K for ISA type E, K, and T low-temperature thermocouple materials. These tables have been smoothly joined at 273.15 K to previous NBS high-temperature thermocouple tables.

Properties of Solid Parahydrogen.—Dielectric-constant measurements along the melting line of solid hydrogen at pressures between 18 and 320 atm and temperatures between 14.4 and 22 K have led to the first calculation of the molecular polarizability (with an estimated precision of a few parts per 10,000) of this substance. New measurements of the melting pressure were also obtained leading to accurate densities for the melting solid. Knowledge of these properties is required for the design of efficient aerospace systems utilizing slush hydrogen (liquid-solid mixtures).

Thermodynamic and Transport Properties

Specific Heats of Gaseous and Liquid Oxygen.—The specific heats, C_v and $C_{\text{saturation}}$, of gaseous and liquid oxygen have been measured from the triple point to room temperature at pressures to 350 atm.

They were combined with earlier PVT measurements to produce what is believed to be the most accurate and comprehensive thermodynamic functions for this substance in the cryogenic temperature range. Accurate thermodynamic functions are required by the aerospace and gas separation industries.

Refractive Index of Gaseous and Liquid Hydrogen.—The refractive index of liquid hydrogen has been measured with great precision over a very wide range of temperature and density. Data were obtained at temperatures between 15 and 300 K and at pressures up to 233 atm using a Fabry-Perot interferometer. The density and temperature dependence of the Lorentz-Lorenz function was also analyzed. The measurements are useful for the determination of fluid densities and mass flow rates, and for providing new information on the interactions between molecules.

High-Speed Thermodynamic Measurements System.—The first phase of the High-Speed (millisecond time resolution) Thermodynamic Measurements System developed for research at high temperatures (1800 K to melting points) has become operational. This unique system provides simultaneous dynamic measurement of specific heat, electrical resistivity, and hemispherical total emittance of electrical conductors. Recording of the data is completely automated; up to 2000 data words can be measured and stored in digital form (with 0.01 percent resolution) in the core memory during an experiment of subsecond duration. This information is retrieved immediately after the experiment and processed by a remote-control, time-sharing computer. Data on molybdenum in the range 1800–2800 K have been obtained.

Quantum Mechanical Virial Coefficients of Helium.—The velocity of sound in helium from 2 to 20 K obtained at NBS has been analyzed and preliminary values of the second virial coefficient in this range have been obtained. These values are better than previous ones and are of importance to improvement of the International Temperature Scale. It was possible to obtain these improved values because accurate theoretical computations of the second virial for the Lennard-Jones potential have yielded an appropriate form for the temperature dependence of the second virial. The calculations covered the complete temperature range from near absolute zero to the classical region. By separating the virial into Boltzmann and exchange terms, the suppression of statistical effects with rising temperature could be examined in detail. The theoretical results have been compared with experiments over the whole range leading to improvement of the intermolecular potential function for helium.

PVT Measurements in the Glassy State.—As part of a continuing study of the influence of pressure, temperature, and volume history on the physical properties of glass-forming liquids and polymers, specific volume measurements to 1000 bar along various isothermal and isobaric paths into the glassy region on poly (vinyl acetate) have been completed. These studies have helped to resolve several controversial points about the formation of glassy state, and the applicability of reversible thermodynamics to the glassy state.

Theory of Transport Phenomena.—Such phenomena as frequency dependence of sound velocity, nonlocal effects near the critical point, and nonlinear dependence of viscosity on the rate of shear are not adequately explained by classical fluid dynamical theory, but require a generalization of it. One generalization, which describes noninstantaneous, nonlocal, and nonlinear responses of flows to the thermodynamic forces (gradients), has been derived by statistical mechanical methods. The conservation equations are unchanged in form, but new expressions are given for the pressure tensor and heat current vector. The new expressions are determined by microscopic quantities (e.g., interparticle potential), and are the sum of reversible and irreversible parts. The reversible parts are the average fluxes in a local equilibrium ensemble that includes nonlocal effects. The expression for the irreversible parts are the analog of the classical transport relations, and are linear combinations of integrals over space and time of the thermodynamic forces multiplied by the correlations of fluxes averaged in a local equilibrium ensemble.

Theory of Critical Phenomena.—A fluctuation theory of critical phenomena in fluids has been formulated and applied in several instances. The theory generalizes the Ornstein-Zernike theory of density fluctuations to fluctuations of energy, pressure and similar functions. It introduces an important new mathematical object, the critical eigenvector, which has several significant roles near the critical point. The difference in molecular distribution functions in the liquid and gas phase just below the critical point is proportional to this eigenvector, and it is the most probable fluctuation in the molecular distribution functions just above the critical point. It is known from experiments that the specific heat at constant volume, while apparently infinite at the critical point, approaches infinity much more slowly than the specific heat at constant pressure. This fact can be explained if the molecular distribution functions have a certain asymptotic behavior, which can be understood from the role of the critical eigenvector as the most probable fluctuation.

The Intermolecular Potential and Experimental Data.—A method to evaluate quantitatively the relationship between model intermo-

lecular potential functions and macroscopic experimental properties has been developed. The method is valid for any potential function and any property, provided the appropriate theoretical expressions are available. A principal conclusion of this work is that a temperature range exists where the experimental properties are insensitive to the potential function. New results concerning the experimental precision required in order to determine the potential function from these properties have also been achieved.

Light Scattering.—The change in the critical temperature of a binary mixture when an external electric field is applied to the fluid has been studied by determining the change in the intensity of the light scattered by concentration fluctuations (critical opalescence) as the field is turned on. A theoretical description of the change with time of the concentration fluctuation correlation function was made which relates the intensity of scattering to the binary diffusion coefficient. The theory enables the binary diffusion coefficient of the mixture to be determined from these measurements.

Critical Point Scaling Law.—The validity of the scaling law equation of state has been tested by an analysis of the data for the critical regions of CO_2 , Xe, ^4He and N_2O . This theoretically based equation of state expresses the nonanalytic behavior of properties in the critical region through numerical exponents and a function, $h(x)$, of a single variable which measures the difference from the critical point. The analysis yielded values of the critical isotherm exponent and the critical temperature based on a proposed form for $h(x)$, satisfying the scaling law requirements. Taking experimental values for the coexistence exponent, a value of the critical isotherm exponent equal to 4.6 ± 0.1 was found to give a good fit for three classical substances, and a value of 4.45 ± 0.15 gives a good fit for ^4He . For all substances, a value of about 4.4 for the ratio of the compressibility along the critical isochore to that along the coexistence line is found. Although this is much smaller than previously reported values, it agrees with the value 4.9 for xenon determined by optical techniques.

Theory of Shock Wave Propagation in Crystals.—Shock compression experiments on solids provide a means for studying the interatomic forces under high pressure (to 10^6 bars) and high temperature (to 10^4 K) conditions. A theoretical study has been completed in which the relaxation process behind a shock front in a crystalline solid has been modeled on a computer. This study provided information related to the interpretation of experimental data from this process in terms of interatomic forces, and on the PVT relationships in the shock compressed region.

APPLIED MATHEMATICS

The Institute for Basic Standards conducts a program in applied mathematics and statistics to meet varied needs in the development of new measurement techniques and in the evaluation of the results of measurement. The level of the mathematics involved makes it essential to conduct fundamental mathematics research on a fairly broad scale.

Routing for Maximum Flow.—A problem frequently arising in operations research studies is that of routing so as to obtain a maximum flow from a “source” to a “sink” in a network whose individual links have limited flow capacity. The standard method of solution of this problem does not generally work if the various link capacities are incommensurable, and, if ratios of link capacities have large denominators, a very great many computational steps may be required. It was found that a slight and natural additional specification to the algorithm enables one to obtain an upper limit on the computation labor which is actually independent of the link capacities, namely, the cube of the number of network nodes.

Mathematical and Statistical Tables and Handbooks.—A sixth printing of Applied Mathematics Series 55—the Handbook of Mathematical Functions—was released in November 1967. With the sale of the hard cover volume approaching 82,000, changes were submitted for the seventh printing to be released in July 1968. A second edition with additions and corrections of “Tables Relating to Mathieu Functions,” initially printed by the Columbia University Press, was released in August 1967.

Unified Treatment of Multidimensional Contingency Tables.—An investigation of interaction in multidimensional contingency tables has led to a unified treatment from an information-theoretic approach. Under this approach the principle of minimum discrimination-information is proposed and used to generate hypotheses concerning second-order and higher order interactions. It is shown that all classical hypotheses for contingency tables can be generated through the use of this principle when certain marginals are considered fixed.

Behavior of Statistical Procedures.—The Bureau’s Statistical Engineering Laboratory performed an extensive analysis of the properties of Cochran-type tests for homogeneity of variance when the underlying distribution is not normal. Numerical results for small sample sizes show that statistical procedures of this type can be seriously misleading if used uncritically when the underlying distribution is not normal. For example, the sample range exhibits behavior different from that predicted by the known approximations based on the fourth

moment when the underlying distribution is sufficiently discrete (as often occurs when measurement data are severely rounded).

Matrix Representations of Groups.—A useful, comprehensive set of notes of Matrix Representation of Groups was completed. The notes serve as a complete introduction to the subject and demand only a knowledge of elementary matrix theory from the reader. An appendix on the elements of the theory of algebraic numbers is included.

Heat and Mass Transfer.—Significant work was performed on the numerical solution of problems in coupled heat and mass transfer, with nonlinear reaction kinetics in which the flux equations are based on the postulates of irreversible thermodynamics. A scheme for integrating the diffusional contribution to the transfer process was developed which effects economy of solution through permitting the use of the maximum time step in each of the coupled equations.

Numerical Analysis.—New methods were developed for checking the reliability of the results of numerical calculation. A new theory of error bounds for Laplace's method of approximating definite integrals containing a large parameter has been developed and illustrated by numerical examples. In addition, the theorem of Piatetsky-Shapiro on quadrature error bounds was extended and sharpened. Further, important new results were obtained on the computation of solutions of second-order linear difference equations. Comparison theorems were constructed which yield strict and very realistic bounds for the truncation error associated with the new method of computation.

TECHNICAL ASSISTANCE TO OTHERS

Studies for Other Groups

Fuel Oil Measurements.—A measurement service extended to the Commerce Department's Maritime Agency has resulted in a significant saving of time and money. The Chairman of the Maritime Administration's Trail and Guarantee Survey Boards has expressed his appreciation for the rapid determination of the heating value of fuel oil which enabled M.A. to advise a steamship company and a shipbuilding corporation that economy trial reruns of a new \$15 million cargo-passenger vessel would not be required. The direct saving to the shipbuilder was about \$10,000, but significant other cost savings in time and equipment were also realized.

Commercial Tungsten-Rhenium Type Thermocouples.—The performance of commercially available W-Re type thermocouples at temperatures up to 2300 °C has been studied, and existing commercial

temperature-emf tables for the thermocouples have been evaluated. The investigation was performed for the American Society for Testing and Materials and will assist in the industrial standardization of these thermocouples. W versus W-26 percent Re, W-3 percent Re versus W-25 percent Re, and W-5 percent Re versus W-26 percent Re thermocouples from two leading suppliers were investigated and their temperature emf relationships were found to conform with the suppliers' temperature-emf tables to within the equivalent of about 1 percent of the indicated temperature for the range 400 to 2100 °C.

Noise Control Guide.—Under the sponsorship of the Federal Housing Administration, NBS has completed an extensive investigation of noise in multifamily dwellings. This investigation resulted in the publication by FHA of a 421 page report entitled, "A Guide to Airborne, Impact, and Structure-borne Noise Control in Multifamily Dwellings." Some 345 detailed architectural drawings show proper construction and installation of wall and floor assemblies, building equipment, appliances, heating, air conditioning, plumbing, and electrical systems required for adequate noise control and privacy. The guide presents recommended sound insulation criteria for three grades of multifamily dwellings and contains sound insulation data, fire ratings, and descriptive illustrations for 137 walls and 111 floor-ceiling constructions.

Fluorescent Orange To Protect Hunters.—The state of Massachusetts requires a hunter to wear "daylight fluorescent red or orange" garments lest he be mistaken for game by other hunters. One fatal accident which occurred two years ago involved a man wearing an inconspicuous, slightly fluorescent, copper-colored vest, legal under the law. Inquiries from several States have indicated a desire to revise present laws by including color tolerances tight enough to assure that the fluorescent orange worn by the hunter is sufficiently conspicuous to afford him adequate protection. Samples of the fluorescent cloth have been measured and recommendations issued for color tolerances which can be met by currently available cloth.

Electroluminescent Formation Lights.—Extensive development for the Naval Air Systems Command has resulted in the use of flat, semi-flexible, strip-type electroluminiscent (EL) lamps as formation lights on military helicopters. Incandescent lamps proved to be infeasible for this application chiefly because of the severe vibrational environment. The linear geometry of the EL lamps has proven to be an added advantage. Studies have begun to determine the feasibility of extending this type of lighting to high-performance (mach 2) aircraft.

Microfilm Preservation.—Since microfilm is one of the most suitable media for the storage of records of permanent value, NBS continually investigates factors affecting the formation of blemishes on such films. Chemicals evolved by the cardboard cartons in which the films are stored may react with image silver to form blemishes. Procedures have been developed for testing the susceptibility of processed microfilm to blemish formation and for testing the relative blemish inducing potential of various carton materials. The effects of photographic processing and storage conditions are being established by these techniques.

Liquid Hydrogen Bubble Chamber Consultation.—Continuing consultation and advisory services were provided on three of the Nation's large liquid hydrogen bubble chambers. Assistance has been given to the Atomic Energy Commission on the planning and cryogenic design of the 12-ft chamber at the Argonne National Laboratory and to the Brookhaven National Laboratory on the planning and safety analysis of their proposed 14-ft chamber. Assistance was also given on the redesign and testing of the 500-liter bubble chamber at the Massachusetts Institute of Technology. The latter chamber was earlier determined to have initiated the fire and explosion at the Cambridge Electron Accelerator that resulted in the destruction of the Experimental Hall.

Studies of Explosives.—A study has been made for the Naval Ordnance Laboratory of the heats of formation of stilbene and hexanitrostilbene. The latter material is a solid explosive analogous to TNT (trinitrotoluene) but having less sensitivity and a very low volatility. This material is being investigated for possible extraterrestrial applications.

Holmium Total Neutron Cross-Section.—A collaborative effort with experimenters at the Atomic Energy Research Establishment, Harwell, England, and theoreticians at the Oak Ridge National Laboratory has been undertaken to measure the total neutron cross-section of oriented ^{165}Ho in the energy range 2 to 135 MeV. The experiment involved cooling a ^{165}Ho single crystal to 0.32 K in an NBS ^3He refrigerator and measuring the neutron spectrum generated by the Harwell synchrocyclotron with a time-of-flight spectrometer. The differential cross section of the oriented ^{165}Ho oscillated in sign between the measured energy limits in a manner consistent both with the nuclear optical model and with a model based on the Ramsauer effect.

Conferences and Symposia

Thermometry Committee Meets at NBS.—NBS played host to the Advisory Committee on Thermometry to the International Bureau of Weights and Measures for the first half of its 1967 meeting on September 5–8. The meeting was divided between NBS Gaithersburg

and National Research Council, Ottawa. Delegates from Australia, Britain, Canada, France, Japan, Netherlands, Spain, U.S.A., U.S.S.R., and West Germany attended. In both institutions a day of laboratory visits was scheduled to provide a break in the discussions which occupied a total of 41½ days. Agreement was reached on the revision and extension of the International Practical Temperature Scale to be effected probably early in 1969.

Symposium on Wolf-Rayet Stars.—During the week of June 10–15, 1968, the American Astronomical Society, Harvard College Observatory, the NBS Joint Institute for Laboratory Astrophysics, and the Smithsonian Astrophysical Observatory sponsored a working Symposium on Wolf-Rayet Stars. It was held at the JILA facility in Boulder, Colo., and was attended by some 45 persons actively interested in the field. This class of star may make significant contributions to understanding the phenomena that condition the state of other stars. They are thus of particular interest. A focus for the meeting concerned the effect of mechanical input into the extended atmosphere. The case of the Wolf-Rayet stars appears to be one for which the whole line spectrum, in the visual as well as in other regions, requires a model departing from wholly radiative effects. The Wolf-Rayet stars probably represent the most extreme case of coupling between aerodynamics and astrophysics to study the behavior of a hot gaseous atmosphere.

Colorimetry and Spectrophotometry Seminar.—Colorimetrists and spectrophotometrists from university, industry, and Government laboratories participated in a 3-day seminar. Topics covered were: psychophysics of color vision, uniform color space, color-order systems, spectrophotometry standards, photodetector response calibration, photoelectric colorimeters, metamerism, and variability of color measurements. Attendees had the opportunity to visit the NBS colorimetry and spectrophotometry laboratories and to discuss special problems with Bureau experts.

Length Measurement Seminar.—Dimensional metrologists from both industrial and Government standards laboratories participated in a 5-day seminar. Topics covered were interferometric absolute length measurements of gage blocks and diameter; interferometric methods for calibration of flatness, surface texture, length, and angle; optical and mechanical methods for the calibration of surface planeness and angle. Experimental and statistical programs for determination of errors were discussed. Attendees had the opportunity to make measurements on NBS equipment.

Seminar on Time and Frequency.—Thirty-six registrants from private industry and several Government agencies attended a 3-day

seminar on Frequency and Time given by the NBS Time and Frequency Division during February 28–March 1, 1968. The seminar included 2 full days of classroom lecture by various experts in the Division and several laboratory demonstrations of state-of-the-art equipment and measurement techniques. The latter included demonstrations of frequency stability measurement techniques, an actual global time synchronization of atomic clocks to about $4\mu\text{s}$, and NBS timekeeping techniques utilizing efficient weighting of individual clocks. One full day was devoted to a visit to the NBS standard frequency broadcast stations at Ft. Collins, Colo.

High Frequency and Microwave Attenuation Seminar.—A seminar in attenuation measurements was presented during March of 1968 for eighteen people from Government and industry. The seminar included definition of terms, discussion on attenuation standards, attenuation measurement techniques, and measurement errors. The lecture sessions were supplemented with laboratory demonstrations.

Microwave Calibration Workshop.—A workshop on Microwave Calibration Services was conducted in May 1968. Forty-six persons attended from industrial and Government laboratories. The attendees were primarily technicians and engineers who are practicing metrologists. The workshop presentations were almost evenly divided between lectures and demonstrations, and covered the subjects of frequency, impedance, attenuation, phase shift, power, and noise.

Precision and Accuracy in Measurement and Calibration.—At a 4-day seminar, thirty-three participants representing 23 industrial and 10 Government standards laboratories obtained firsthand information on some of the statistical techniques in current use at the Bureau. The Bureau reported experience in the development and use of statistical experiment designs especially tailored to the needs of calibration laboratories for making economical intercomparisons among sets of standards. Presentations by NBS statisticians were complemented by reports from Bureau calibration laboratories that are implementing the new computer-aided methods for data analysis, preparation of calibration reports, and surveillance of the measurement (“production”) process.

Seminars on Statistics.—A series of 15 expository seminars for chemists and physicists was conducted on the application of statistical techniques in the design, analysis, and interpretation of scientific data. The series, planned jointly by the NBS Applied Mathematics and Analytical Chemistry Divisions, served as a useful forum for consideration of the nature and value of the methods of statistics in scientific investigations.

INSTITUTE FOR MATERIALS RESEARCH

Emphasis in the Institute for Materials Research (IMR) is on materials measurement methodology with particular concern for the characterization of materials. This emphasis supports the unique mission to develop standards of measurement for the commercial, industrial, and educational communities of the Nation and to provide research and consulting services to other Government agencies. About 25 percent of IMR's work is for other Government agencies.

Work falls into the following broad areas:

1. Preparation and Characterization of Materials
2. Data on the Properties of Materials
3. Technical Assistance to Others.

PREPARATION AND CHARACTERIZATION OF MATERIALS

This area includes such activities as the synthesis of new materials, purification of materials, crystal growth, and the analysis of materials with respect to chemical composition and structure. Outputs of these activities are sample materials to be used for specific research purposes, and the development of new techniques and procedures for preparing or characterizing materials. Another output is a class of materials called Standard Reference Materials. These are prepared and sold by IMR and are used mainly by industry for the calibration of equipment.

Preparation

Synthesis

Synthesis of Alkaline-Earth Transition-Metal Oxides.—Oxide compounds of the alkaline-earths and elements of the first transition series have been investigated at elevated temperatures and controlled oxygen pressures. Of primary interest is the influence of stoichiometry on compound formation, stability, and crystal structure which ultimately may dictate the electrical and magnetic properties of the materials. Phases in the systems SrMnO_{3-x} , BaMnO_{3+x} , and $\text{BaFe}_{1-y}\text{O}_{3-x}$ reflect the importance of varying oxygen content (transition metal valence disproportionation) and/or transition metal concentration.

Reactive Intermediates in Chemical Synthesis.—Chemical transformations of inorganic compounds under energetic conditions have been explored from the viewpoint of applications to synthesis of new

materials. Studies of the chemistry of inorganic fluorides in microwave discharges have demonstrated the synthesis of a number of new oxyfluorides of boron, silicon, germanium, and phosphorus, and have yielded information on the reactions of oxides and of polymers with reactive molecular species. A significant chemical discovery is the observation that the complex chemical events in the discharges can be rationalized in terms of surprisingly few intermediates, conventional reaction mechanisms, and chemically reasonable structures. The reactive intermediates produced in these discharges, or by thermal or photochemical excitation, represent novel chemical reagents that are difficult or impossible to obtain by other means.

Synthesis and Crystal Structure of a Trinuclear Complex.—The complex *bis*[triaquo-tri- μ (1,2,4, triazole ($-N_1-N_2$) nickel] nickel hexanitrate dihydrate has been synthesized from an aqueous solution of $Ni(NO_3)_2$ and 1,2,4 triazole. Its crystal and molecular structure determination revealed that the material has a trinuclear complex cation and is monoclinic. The structure was solved by the Patterson and electron density Fourier methods. The material is one of a class of inorganic compounds which contain two or more paramagnetic metal ions per molecule. Such polynuclear species are well suited to detailed study because the exchange coupling between paramagnetic ions in a single molecule is greater than that between ions from neighboring molecules. Therefore, the ions within a molecule behave as magnetically isolated clusters.

New Luminescent Materials.—Yttrium disilicate, $Y_2Si_2O_7$ (yttrialite), occurs in both low- and high-temperature forms in nature. High-temperature yttrialite, in the form of a fine powder, has been synthesized from precipitated gels in air at 1300 °C. When doped with 5 to 10 mol percent of europium or terbium, the material shows intense luminescence when excited by ultraviolet light.

Gadolinium disilicate, a compound isostructural with high-temperature yttrialite, shows very high luminescence when doped with 10 mol percent europium, the luminescent intensity stronger than that of yttrium disilicate containing europium.

Yttrium may be replaced by cerium in $Y_2Si_2O_7$ under hydrothermal conditions at 700 °C and 1 kbar. Up to 60 percent replacement of Y^{+3} by Ce^{+3} is possible without inducing phase transformations. $Y_2Si_2O_7$ containing 25 mol percent cerium shows a very intense bluish-white luminescence. The intensity decreases progressively with increasing amounts of cerium.

New Method for Esterification.—Esterifications are among the most commonly employed and important reactions of synthetic organic chemistry, and many methods have been devised for effecting such

reactions. Nevertheless, there are enolic compounds, for example, that have not been successfully esterified by any of these techniques. Difficult acylations can now be achieved by a novel reagent combination, an anhydride (acetic, propionic, butyric, etc.) dissolved in 100 percent syrupy phosphoric acid. Products are isolated by treatment with ice water or directly by extraction. In this way, croconic acid, which has resisted all other methods, is converted into a crystalline diacetate derivative. Other compounds, easily acylated by other methods, were obtained for the first time as crystalline products.

High Pressure Polymerization.—Pressures of 10,000 to 20,000 atm (about 10 to 20 kbar) have been used to convert fluoromonomers to new polymeric materials having excellent physical properties combined with thermal and oxidative stability. Gamma rays are often used to initiate these reactions since they readily penetrate the thick-walled steel vessels used to create the high pressure. New polymers prepared include polyperfluorostyrene, poly-3,3,3,2-tetrafluoropropylene, poly-3,3,3-trifluoropropylene, polyperfluoroheptadiene-1,6 and various copolymers of the monomers of these polymers with tetrafluoroethylene.

Crystal Growth

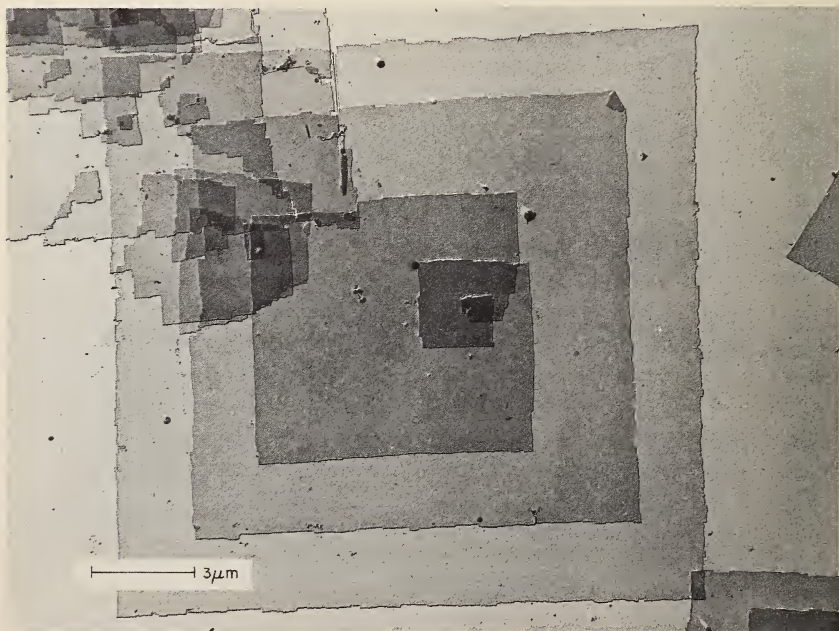
Wide Solid-Solution Series of Apatite Compounds.—Crystals of rare-earth fluoro-silicate apatites and oxyapatites of wide variations in chemical composition were grown. These crystals are of interest for detailed structure analysis of apatite, a stable phase with an exceptional tendency to accommodate cations and anions of diverse shapes and sizes. Specific practical applications are not obvious, but apatites are potentially useful laser hosts and could also be used for high temperature applications.

Sodium fluoride was found the most effective flux for growing the fluoro-silicate apatites, and sodium tungstate is useful for silicate oxyapatites. Growth by isothermal evaporation at high temperature, relying on spontaneous nucleation in 25 ml platinum crucibles, produced transparent crystals of $\text{Na}_2\text{Ln}_8\text{Si}_6\text{O}_{24}\text{F}_2$ (apatite, $\text{Ln}=\text{La}$, Pr , Nd , Sm , and Gd) up to 8.0 mm in size with well developed faces. The colors of the crystals were characteristic of each type of trivalent rare-earth ion. Unit-cell dimensions of all analogs were determined. Europium and terbium doping of the gadolinium analogs produces intensely luminescent crystals.

Crystallization of Ice.—Crystallization processes do not usually occur by the propagation of an interface of single shape. However, if the crystallization process is carefully controlled, as for example in crystal growth, a simple interface shape can be retained.

A recent experiment has provided experimental confirmation of current morphological stability theory. Cylindrical crystals of ice of known orientation were immersed in a bath of slightly supercooled water. The crystal remained smooth during the initial stages of radial growth, but eventually developed undulations. The growth rates of these undulations were in good quantitative agreement with morphological stability theory.

Growth of Curved Crystals.—Studies of the growth of polymer crystals from solution have shown several mechanisms by which the direction of growth is rotated out of the plane of the nucleus. If crystals are grown from very dilute solutions at small supercoolings, relatively simple and planar crystals retaining the symmetry of the unit cell result. For poly(4-methylpentene-1), as the solution concentration and supercooling is increased, relatively more complex and curved crystals result. The curvature, which is a characteristic common to several polymers, results from the combined effect of strain caused by the molecular folds on the crystal surface and the subdivision of a crystal into different fold domains. Such effects no doubt play a role in the growth of structures with spherical symmetry (spherulites) which dominate the morphology of melt crystallized commercial polymers.



Simple planar crystals of poly(4-methylpentene-1) grown from very dilute solutions at small supercoolings. Studies on such crystals have shown several mechanisms by which the direction of growth is rotated out of the plane of the nucleus.

Electrodeless Electrolysis.—An electrodeless electrolysis process was developed in which electrolysis takes place in a glow discharge or a stream of electrons in vacuum rather than at the surface of the metallic electrode. Silver chloride was used to produce a silver coating and a fused nickel salt was used to produce nickel powder. The principal purpose was to further confirm the theory of electrolysis; however, metal coating ceramics may become practical.

Characterization

Composition

Limits for Qualitative Detection and Quantitative Determination.—

Because of the numerous, inconsistent and restricted definitions of a lower limit of detection in analytical chemistry, IMR examined the statistical theory of signal detection and signal extraction. A series of principles were evolved for unambiguously describing the sensitivity of an analytical technique. Limiting sensitivity levels were defined for (1) L_c which is the critical level below which a signal cannot be detected with a given probability, (2) L_d , the a priori detection limit, which together with L_c determines the probability of a correct detection, and (3) net signal limit which permits a statement of magnitude in addition to detection.

New Coulometric Method for Boron Determinations.—Boron is commonly determined by conversion to boric acid which is acidimetrically titrated in the presence of mannitol. A new method has been developed which carries out this titration coulometrically, resulting in an accuracy of better than two-thousandths of a percent. This is a factor of ten better than that obtained by conventional procedures. The method is especially useful for, and has been applied to, the characterization of primary standard boric acid. Because only small samples are required, it is very advantageous for highly accurate microchemical analysis. The new method is one of a series of coulometric procedures developed in IMR; each of these has a precision and accuracy about ten-fold greater than those of the most refined conventional analytical techniques.

New Neutron Generator Facility.—A new neutron generator has been installed and the radiation shielding and pneumatic tube transport system have been upgraded to make this facility one of the finest of its kind. Using this new generator, methods have been devised for the evaluation of systematic errors due to differences in self-shielding of neutrons and self-absorption of gamma rays between the sample and the standard. The system was calibrated with samples of known density and composition, and by using the correction factors generated, the element of interest may be determined accurately in any matrix

material. The general technique can be routinely applied to both trace and macro elemental determinations.

Using this facility, analyses have been performed for the determination of oxygen in metals and for metal in the metallo-organic compounds with accuracy and precision comparable to many non-nuclear methods.

A-C Scanning Derivative Apparatus for Spectrometry.—An apparatus based on an a-c scanning derivative principle has been developed which should greatly enhance the potentialities and usefulness of flame emission and atomic absorption spectrometry. The new apparatus, which consists of a vibrating mirror or quartz plate operated at controlled frequencies, permits the repetitive scanning of a narrow region of spectrum and in so doing offers significant improvement in the precision and accuracy obtainable in both the emission and absorption modes.

Heretofore, a serious drawback in atomic absorption spectrometry has been the need to use a separate source for each element. The use of a continuum source has been restricted because such systems require some means for modulating or chopping the light beam as well as a higher resolution monochromator. The a-c scanning technique minimizes the need for high resolution instrumentation. This modification should not only reduce the cost of instrumentation but should also provide impetus for future development of multichannel methods by which a large number of elements can be determined with a single source.

The advantage of the new technique has been demonstrated by the successful determination of several elements in the presence of elements which normally interfere when examined in the usual manner. For example, barium can be readily determined in the presence of 1000-fold excess of calcium. Lithium can be determined in sodium at less than 0.01 part-per-billion level.

This development should facilitate the determination of trace elements of importance in new materials and biological or environmental problems.

Activation Analysis With the NBS Reactor.—The 10 MW nuclear reactor at NBS began low-power operation in December, 1967, and will achieve full-power operation by November, 1968. Facilities have been constructed for the irradiation of samples in the reactor for periods of hours or days using a special sample suspension system through the top shield plug of the reactor. Rapid (less than 2 s.) pneumatic transfer facilities from various locations in the reactor core and from the graphite thermal column allow the experimenter to choose widely varying ratios of "fast" to "thermal" neutrons from a pure thermal neutron spectrum in the thermal column facility to about 60 percent

greater-than-thermal neutrons in the reactor core. A "fast neutron converter" facility is in the development stage; it will permit irradiations with pure fission spectrum neutrons without thermalization. By choosing these ratios carefully, the experimenter can maximize a desired nuclear reaction, or minimize a competing reaction.

High-Precision Flame Emission Spectrometry.—A new technique of differential flame emission spectrometry was developed whereby a precision of one part in a thousand was obtained over a concentration gradient of a few parts per million to several percent. This 20 fold improvement in precision is equivalent to or better than the present classical chemical methods. To accomplish this improvement in precision, the various instrumental parameters were optimized. This technique was evaluated by the determination of 10 percent lithium in glass beads which are to be used for neutron flux measurements. The same approach can be applied to any analysis in flame emission or atomic absorption spectrometry and in any matrix such as biological materials, and ferrous or nonferrous metals.

Trace Quantities of Yttrium in Rare Earths.—Trace quantities of yttrium (Y) in other rare earths were determined with a ± 5 percent precision at the 20 ppm level by utilizing the photonuclear reaction $^{89}\text{Y}(\gamma, n)^{88}\text{Y}$. An analysis based on coincidence spectrometry utilizing gamma ray decay rather than the more conventional analyses based on chemical reactions was necessary because of the close similarity of the chemical properties of the rare-earth elements. Nuclear activation analysis utilizing the ^{90}Y isotope produced by neutron bombardment of the stable, naturally occurring isotope ^{88}Y also was not utilized because of the lack of gamma rays associated with the ^{90}Y decay.

Identification of Unstable Clinical Standard Compounds.—IMR chemists have devised new methods of measuring the purity of a series of unstable chemical compounds that are particularly significant to clinical calibration and measurement. Ordinarily, the analysis for chemical purity is long and difficult when applied to compounds such as urea, D-fructose, and uric acid, which are known to undergo decomposition if they are improperly treated. To avoid this problem and yet obtain accurate chemical measurements, the method utilizes formation of a chemical derivative that is much more stable than the original compound.

Of particular importance is the technique of trimethylsilylation which, when applied to the above compounds, yields derivatives that are stable as gases when heated. These gases are then analyzed by gas chromatography which serves to discriminate one compound from another. Of even greater importance is the fact that the technique permits separation of various contaminants which can then be fed into

a mass spectrometer and in a very short time their molecular weights determined. The techniques of isolation and characterization can be applied to as little as 10^{-9} g.

Carbon Isotope Analysis.—A method has been devised for measuring the C^{13}/C^{12} ratio in C^{13} enriched samples by combining gas chromatographic separation techniques and analysis by means of a sensitive, differential gas density balance. A C^{13} enriched sample of an organic compound is burned to CO_2 and H_2O , the water separated out on a gas chromatographic column, and then the density of the enriched CO_2 compared to that of normal carbon dioxide. The minimum detectable C^{13} enrichment is about 0.02 percent of the natural abundance which suggests that the method will be applicable to geochemical analyses. The technique is also applicable to the determination of stable isotopes other than those of carbon.

X-Ray Powder Patterns—Saline Series Standards.—In recent years there has been a renewed interest in the recovery of a variety of commercial products from saline residues. More than one hundred compounds may be encountered in exploring various methods of separation of these salts. Identification of these anhydrous and hydrated compounds is best accomplished with x-ray diffraction. Because this method of analysis is dependent upon the availability of reference patterns, IMR has added over 50 new standard reference patterns of saline compounds to several that were originally available in the Powder Diffraction File. In the next 2 years, another 50 such patterns will be added in an effort to present an extensive ocean-salt series for those who work with solids that accumulate from desalinization operations.

Nuclear Magnetic Multiple Resonance for Molecular Structure Determination.—Development of double-resonance techniques to a routine adjunct of high-resolution nuclear magnetic resonance measurements greatly enhance the power, utility, and versatility of n.m.r. in the determination of molecular structure. This has been demonstrated in IMR laboratories by measurements of n.m.r. parameters for the silicon-29 nucleus in a number of inorganic and organic silicon compounds. The silicon spectra, which are observed indirectly, frequently contain structural information not present in the commonly observed proton or fluorine n.m.r. spectra of these compounds. The method is being applied to a wide range of compounds of other nuclei such as tin, lead, and mercury.

Electron Microprobe Color Photography.—A procedure for the color representation of electron microprobe area-scan images has been developed. The net result is a means of showing microstructure in terms of specimen composition. In order to obtain the color composite, x-ray

area-scans are prepared as positive black-and-white prints by photographing the cathode ray tube of a scanning electron probe micro-analyzer. This procedure indicates the location of a particular element in the microstructure by showing a bright spot whenever that element occurs. The composite is obtained by combining up to three such photographs using red, green, and blue filters interposed between each photograph and the color film in a copy-camera. If rapid-development color film is used, the process is quite inexpensive. The color method can be applied to any material from which characteristic x-ray line scanning images and electron scanning images can be obtained. IMR has applied the method to archeology, biology, dentistry, failure analysis, metallurgy, meteorites, and mineralogy.

An Automatically Operated Mass Spectrometer.—A two-stage 90° sector mass spectrometer used for isotope ratio determinations has been converted to automatic operation with the addition of a small programmable calculator and suitable interfacing components. With this conversion, samples as small as 10^{-12} g with isotope ratios of more than 500:1 have been analyzed and it is anticipated that samples several orders of magnitude smaller may be utilized in the near future. In addition, the time for an analysis has been materially shortened and the accuracy and precision obtained has equalled that expected with much larger samples. The components necessary for this conversion were the calculator with its teletypewriter printout device, digital control and command converters and a dual digital counter.

With the use of this system the mass spectrometer may be programmed to collect data at predetermined times during a scan of the mass region of interest or, alternately, to repetitively collect data representing the heights of as many as eight mass peaks, to calculate the ratios of these peaks to each other and to readjust the time of data collection to give constant, predetermined, counting statistics.

New Concept of Microstandards.—The problem of isolating and measuring extremely small quantities of matter in the region below one nanogram is of growing importance to modern science and technology. The development of standards containing such small quantities of matter in exactly known quantity is therefore of considerable interest. The need for this is shown by the conspicuous absence of standards for determining detection limits and performing quantitative calibration in the subnanogram region. These quantities are below the limit of weighability.

An important step toward the solution of these problems has been made by IMR chemists. Spherical and chemically uniform beads of ion exchange resin have been loaded with the ions obtained in water from various electrolyte compounds. A technique has been found for isolating individual beads of very small size, weighing to one trillionth



Components (teletypewriter printout device, digital control and command converter, and a dual digital counter) to automate mass spectrometer being used for isotope ratio determinations.

(10^{-12}) gram, in such a manner that all of the ions remain with the bead. The method of "two dimensional dispersion" permits the identification of a single bead with a microscope and various procedures can then be used to measure the particle diameter. Instead of weighing the bead, the weight is computed from the measured diameter, the known chemical homogeneity of the material, and the independently measured density of the beads. A single bead thus attains the role of a standard mass that can be reliably measured. With the use of conventional micromanipulation apparatus it can be transferred to a chosen instrument requiring calibration for the type of matter contained by the bead.

Analytical Chemistry of Corn Syrups.—Recent technological developments in cornstarch hydrolysis have resulted in the industrial production of many new types of syrups that have expanded the use and market for corn—the most valuable single crop in U.S. agriculture. Relationships in the physical properties of corn syrups, developed some years ago to characterize syrups for commerce and use, are now found to be not entirely satisfactory for the new products. Furthermore, international controversy exists regarding the adequacy of some of these relationships. To remedy this, a Research Associate program is being conducted at NBS in cooperation with the Corn Industries Research Foundation in order to evaluate with modern techniques the properties of the broad range of syrup compositions now available.

Fundamental to the reliable measurement of many of the significant properties of syrups is an accurate knowledge of their moisture content. Hence, prior to undertaking the study of other properties, methods for determining water in syrups are being evaluated. The standard industrial method (mixing with filter-aid and drying in vacuum oven) is found to be only slightly inferior in accuracy and precision, but less satisfactory with respect to time required for analysis, to a new spectroscopic method employing the direct measurement of a water band in the near infrared. The much-used Karl Fischer titration is found equally as reliable as the vacuum-oven method. Other methods explored are attenuated total reflectance, thermogravimetry, and colorimetry; these are somewhat less satisfactory, but adequate for use in quality control.

Microanalytical Fluoride Determination.—An analytical method for determination of very small amounts of fluoride has been devised. Two commercial fluoride-sensing electrodes are used with one modified to accommodate $5\ \mu\text{l}$ ($\frac{1}{16}$ of a drop) of solution containing the sample. By comparison with standards, as little as $0.2\ \text{ng}$ of fluoride can be determined with a reliability of 1 relative percent. Since the electrode is highly selective for fluoride ions, this constituent may be determined in rather complex mixtures without separation. Accordingly, the new microchemical technique should be applicable to a variety of analytical problems.

New Method for Trace Analysis of Oxygen in Gases.—A highly sensitive and accurate absolute method has been found for the determination of trace amounts of oxygen in inert gases. In this method, the gas is passed through a galvanic cell with silver and lead electrodes that are covered with a film of potassium hydroxide solution. The current output of this cell depends on the rate of oxygen consumption, which in turn is related to oxygen content and rate of flow of the gas, as well as to certain kinetic parameters.

Because of the time factor, the cell is not 100 percent efficient; hence, the laws of electrolysis cannot be applied as in coulometric procedures. To overcome this problem, the gas is passed successively through several cells until the oxygen is essentially completely reacted. Moreover, the measurement is conducted at two different rates of flow to cancel the kinetic effect. The difference in total current for the two experiments can then be equated to the absolute oxygen content by use of quantitative electrolytic considerations. This method is very simple to use, yet it provides analyses that are accurate to at least 1 part in 100 in the concentration range between 0.1 and 0.0001 mole percent of oxygen.

New Method for Antimony Determination.—The accurate determination of antimony in steel at the 10 to 50 ppm level has presented numerous problems. Using a highly sensitive and selective spectrophotometric procedure, IMR chemists can now determine as little as 0.01 μg of this element. This work was initiated originally by requests from the rotor steel industry to provide new Standard Reference Materials with a certified antimony content. The need was based on metallurgists' belief that trace amounts of antimony were directly responsible for many of the cracking failures occurring in the rotor blades of turbine generators. The only interfering elements in the new procedure are gold and thallium; their presence can be readily discerned, however, and easily eliminated. Following dissolution of the sample in a sealed glass tube at approximately 250 °C, antimony can be determined in the resulting solution in 5 min or less. The expected accuracy is ± 2 to 5 percent of the amount present.

Automatic Grid Scan for Electron Probe Microanalysis.—An automatic system for grid scanning and registration of x-ray counts, installed by Institute scientists for the study of homogeneity of standard reference materials over micro areas, is a major step toward automation of the electron probe microanalyzer. This system provides the hardware for control of line and grid scans over matrices of selectable dimension up to 100 \times 100 points, and readout of up to six numbers which can represent time, x-ray counts, digitized target current, or monitor current measurement data. Output is presented in the form of coded paper tape which can be printed on a teletypewriter, fed into the memory of a multi-channel analyzer, or used for computation on NBS computers.

Improved Design of Clinical pH Electrode.—Glass microelectrodes with tips small enough to be inserted into single cells are furnishing increasingly useful results in studies of human physiology. These electrodes make it possible to measure the concentrations of sodium and potassium and to study the acid-base balance in living organisms. Hence, they offer hope of future clinical applications in the monitoring of pathological conditions. It is often not possible to assure close uniformity of temperature throughout the measurement system comprising glass electrode, reference electrode, and living subject. Hence, some variability due to changes of temperature is difficult to avoid. During the past year, a means was found to reduce or even eliminate this source of error in systems containing pH microelectrodes. This favorable result can be accomplished by careful choice of the inner element and the inner solution from which the electrode is formed. In the most suitable combination, these two components act together to nullify completely the effect of fluctuations in the temperature of

the laboratory or operating room. The arrangement is applicable as well to the reduction of temperature effects in pH equipment used to monitor the acidity of industrial process streams.

Time-Sharing Computations in Spectrochemical Analysis.—The use of computers to facilitate analytical chemical operations has been undergoing a rapid growth. A report prepared by chemists of the Institute for Materials Research will serve as a guide to laboratories desiring to avail themselves of time-sharing computers. The IMR chemists are believed to be the first to use this type of computational facility in spectrochemical analysis; the methods they have developed have elicited considerable interest, and other spectrochemical laboratories have recently installed terminals connected to time-sharing computers.

Measurement Scales for Ionic Activity.—Electrical response of the relatively new ion-sensing electrodes depends on the concentration of a particular sort of ions present in the solution in which the electrode is immersed. Ion-selective sensors have been designed for the measurement of some 10 different ions, both metallic and nonmetallic. They facilitate the rapid measurement and control of ion concentrations which are time consuming and laborious by conventional analytical procedures. Nevertheless, the measured voltage of the ion-selective electrode is not a direct measure of the desired concentration of the ionic constituent but rather of its effective concentration or activity. To enhance the accuracy and usefulness of the measurement, the electrodes require reference standards for ion activity. During the past year, useful approaches to ion-activity standardization were investigated. The practical solution selected bases ion activities on the same conventional numerical scale on which the national pH scale is established. It is now possible to assign standard values of ionic activity to reference solutions and thus to improve the accuracy of ion-selective sensors used for measuring concentrations of single ionic species.

Structure

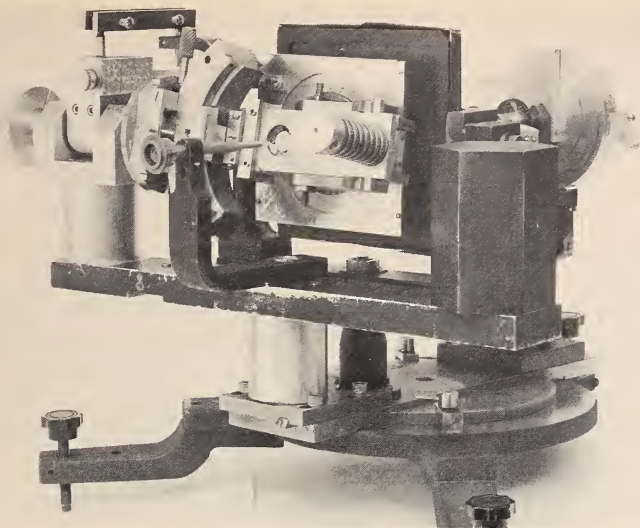
Electron Microscopy Examination of Refractories.—An understanding of the physical and chemical properties of refractories can lead to improved performance of these materials. The properties are known to depend on the manner in which the refractory components are joined together; that is, on the microstructure of the refractory. The microstructure is most commonly evaluated using the optical microscope. However, because of the limited resolution of the optical microscope (1 to 3 μm) many details are lost and an improved technique would be desirable. The Institute for Materials Research has developed a superior method of examination in which thin sections of

refractories are examined by transmission electron microscopy. Specimens of periclase refractory have been examined in this manner and details of the microstructure not capable of resolution in the optical microscopy have been observed. The information so obtained is already being put to commercial use in the development of periclase bricks for the steel industry.

Single-Crystal X-Ray Diffraction at High Pressures.—A diamond-anvil high-pressure cell for use to pressures of approximately 50,000 atm (about 50 kbar) has been constructed using only polycrystalline beryllium as metal components. The cell is transparent to x rays and permits observation of a large fraction of the x-ray diffraction pattern of single crystals of high-pressure phases. The cell is used to determine the unit cell size and symmetry properties of any material whose single crystals can be grown under pressure. This includes material normally liquid at room temperature and at temperatures as high as approximately 300 °C under pressure. Included also are materials which are gaseous under normal conditions with boiling points as low as -50 °C and materials which are solid under normal conditions but have high-pressure polymorphs.

To determine the structure of the material, it is necessary to measure the intensities of the diffracted spots with precision. These intensities are affected by absorption in both the diamond anvils and the beryllium metal of the cell. It is therefore necessary to correct the observed values for this absorption. The appropriate corrections have been calculated for all orientations of the cell and positions of the diffracted spots, and have been verified by measurements on crystalline bromine. Bromine, a liquid at room temperature and pressure, freezes at about -13 °C at 1 bar. Its crystal structure has been determined previously with crystals at low temperature. Using the beryllium cell, bromine was frozen at room temperature and approximately 5000 atm (5 kbar). The symmetry properties and unit cell were found to be consistent with those of the low temperature crystal. The intensities calculated from the known structure were compared with the observed intensities as corrected for absorption in the diamonds and the cell and good agreement was obtained.

Pressure Indicator for Use in the Diamond Cell.—A satisfactory spectroscopic pressure indicator for use in the diamond-anvil high-pressure cell has been developed. The calibrant consists of a spectroscopically thin slab of nickel dimethylglyoxime placed in the pressure chamber between the diamond anvils with the material being studied. The position of the pressure-sensitive absorption band in $\text{Ni}(\text{DMG})_2$ is determined spectroscopically and can be used to define the pressure to within $\pm \frac{1}{4}$ kbar. The calibration curve, determined by the freezing pressures of liquids at room temperature, is rigorously valid only



Crystal structures of materials at high pressure are determined by x-ray diffraction in this assembly. Single crystals, grown in the beryllium diamond-anvil high-pressure cell (center), are oriented in the x-ray beam by means of the special goniometer head (left). X-ray diffraction precession patterns are recorded by the film located at the rear.

at room temperature to pressures of approximately 20 kbar, the freezing point of ethyl alcohol. An additional measurement on methyl alcohol is available but the reported freezing point appears to be uncertain. If a reliable value for the freezing point of methyl alcohol is reported, the range of utility of nickel dimethylglyoxime will be extended to approximately 40 kbar. Although the calibration curve has been determined only at room temperature, it appears most likely that it can be applied at higher (to 100 °C) and lower temperatures by using a linear correction.

Automated Diffractometer for Crystal Structure Determinations.—A single-crystal x-ray diffractometer and ancillary instrumentation has been assembled to operate (1) on-line under direct control of a remote, time-shared computer with communication via a full duplex teletype channel requiring no special interface and (2) off-line under paper tape control. This instrument is used in place of conventional x-ray cameras for obtaining x-ray diffraction patterns needed in determining the structure of crystalline solids. The instrument orients a material sample in an x-ray beam and measures the intensity of the diffracted beam. By eliminating the need for an operator to make the angle settings and transcribe the data, the automated system makes possible around-the-clock operations with increased speed and accuracy. The system differs from others in that it has been designed to operate both by reading a punched paper tape prepared by a com-

puter (off-line control), or by a direct link with a remote computer over a telephone line (on-line control). Crystallographers in IMR now have new freedom in tackling structure determinations of great complexity. The result is likely to be a more thorough understanding of the structure of many more classes of materials.

XRAY67—A Program System for X-ray Crystallography.—Cooperative efforts by scientists in IMR, the University of Maryland, and the U.S. Geological Survey have produced a general x-ray computing system, XRAY67, that includes all the programs required to solve structures using either x-ray or neutron diffraction data. The treatment of different diffraction data is similar with respect to symmetry, settings, amount of data, number of parameters, and ease of use. This system is designed to be used by scientists in laboratories involved in structure determination.

XRAY67 consists of a set of Fortran programs which share a common data file and includes programs that permit: data reduction, the calculation of electron density maps, least squares refinement of atomic parameters, the calculation of bond angles and distances, and many other calculations required in the determination of crystal structures.

Dislocations in Crystals.—Deviations from a perfect crystal structure such as dislocations and impurities are known to affect the strength and other mechanical properties of the crystal. Hence, it is necessary to have an adequate theory of dislocations available for application to real substances. A step in the development of such a theory was the completion of a complex nonlinear dislocation theory for applications to interactions between dislocation groups and to concentrated distributions of dislocations.

Molecular Conformations by NMR.—The conformation (topography) of four 1,2:3,5-diO-benzylidene- α -D glucofuranose derivatives have been determined by nuclear magnetic resonance. The proton spectra of the compounds were recorded from solutions that were maintained in a magnetic field of 2.3 T., corresponding to a radio frequency of 100 MHz. At this high field strength, many of the proton multiplets were resolved.

Proportional Detector for Mössbauer Spectrometry.—One problem that has prevailed in the measurement of Mössbauer spectra is the time required to obtain a high quality spectrum. Proportional detectors are used to detect radiation from the source but they have low efficiency. A new detector has been designed which has a much larger radiation sensitive area than has been used before. Serious difficulties in degradation of detector resolution were resolved by careful positioning and sizing of the electrodes in the detector.

The detector was optimized to detect the x-rays produced by the internal conversion process resulting from the decay of the Mössbauer energy level in ^{57}Fe . This was done by optimum selection of detector thickness and by use of a methane-argon detector gas. This device can also be used to detect conversion electrons by replacing the detector window by a sample to be measured and by using a methane-helium detector gas.

Mössbauer Spectrometry of Nickel Compounds.—The practical application of Mössbauer spectrometry to the study of nickel compounds has been enhanced by the discovery of a practical source of radiation. This source is produced from bremsstrahlung generated by the NBS linear electron accelerator via the $^{62}\text{Ni}(\gamma, p)^{61}\text{Co}$ reaction which decays by beta particle to the 67.4 KeV Mössbauer level of ^{61}Ni . A single line source was discovered by producing a chromium-nickel alloy with composition adjusted to eliminate an internal magnetic field. As a result of the discovery of this source, nuclear data such as the magnetic moment ratio of the excited to the ground state and the nuclear radius change during the nuclear transition have been measured.

The nuclear magnetic hyperfine interaction is large enough in ^{61}Ni to permit the measurement of the internal magnetic fields in nickel compounds and alloys. Although the chemical shift is small compared to the linewidth, it is possible to identify the formal oxidation states of nickel in chemical compounds.

Crystallography of Calcium Carbonates.—Three anhydrous calcium carbonates—calcite, aragonite, and vaterite—are widely encountered; the existence of two hydrated forms, $\text{CaCO}_3 \cdot 6\text{H}_2\text{O}$ and $\text{CaCO}_3 \cdot \text{H}_2\text{O}$ is less recognized. As a result, the possible role of the hydrated salts, particularly in the formation of anhydrous salts, has been largely overlooked. The crystallography of calcium carbonate has been investigated by IMR scientists because of its possible relationship to calcium phosphate mineralization in tooth and bone. The crystal structure of $\text{CaCO}_3 \cdot 6\text{H}_2\text{O}$ was determined and was found to contain discrete ion pairs, $[\text{CaCO}_3]$, surrounded by water molecules. The occurrence of this grouping in $\text{CaCO}_3 \cdot 6\text{H}_2\text{O}$ accounts for the facility with which these crystals grow. This is the first crystal in which this ion pair has been found, and its structure has bearing on the structure of the $[\text{CaCO}_3]$ ion pairs that are believed to exist in solution.

In parallel investigations, the structures of two hydrated calcium sodium carbonates, $\text{CaNa}_2(\text{CO}_3)_2 \cdot 5\text{H}_2\text{O}$ (gaylussite) and $\text{CaNa}_2(\text{CO}_3)_2 \cdot 2\text{H}_2\text{O}$ (pirrsonite) were determined. The structure of gaylussite is of particular interest in that it contains $[\text{CO}_3\text{CaCO}_3]$ ion triplets.

Structure of Water.—It is generally accepted that the structure of water around solutes has an important effect on the thermodynamic properties of aqueous solutions. The forces between protein molecules are greatly affected by these structures. NMR spectra were determined for water absorbed in collagen, the main protein of connective tissue. These spectra show that the water molecules diffuse rapidly through the pores, but do not rotate isotropically.

Polymer Characterization.—Physical properties such as viscosity and plasticity are mainly dependent on the distribution of molecular weights in the polymer sample. Techniques for fractionation have now been developed for polyethylene, the commercially most important plastic at present. Several instruments can be used for molecular weight determinations of the fractions, provided they have been calibrated.

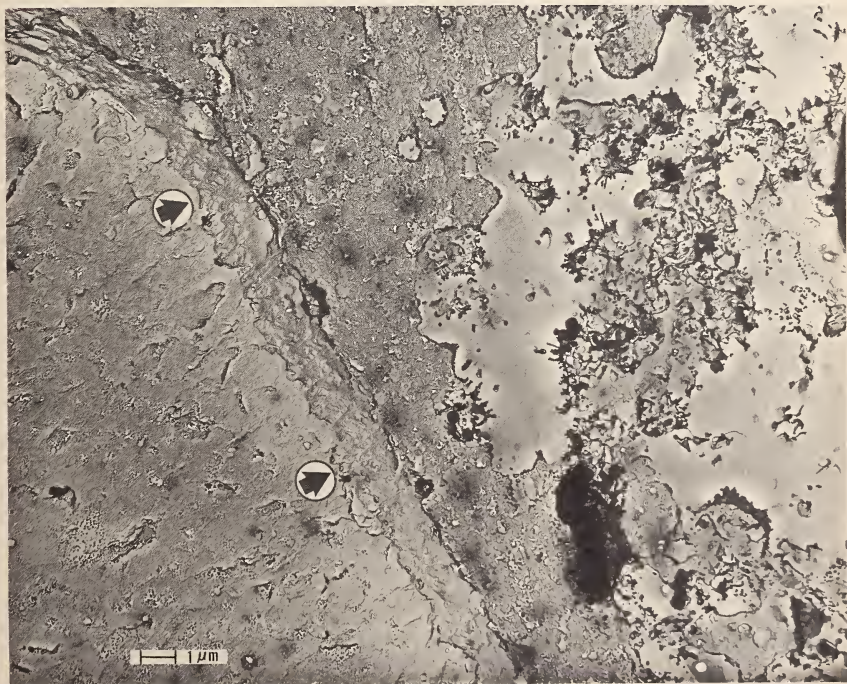
Variation of Unit Cell Dimensions.—It has been shown that the orthorhombic unit cell dimensions of a given polyethylene are not unique, but depend upon the physical history of samples crystallized from either the melt or solution. Variations in cell dimensions result from variations in crystallization temperature, annealing temperature, and annealing time as well as from deformation of the crystals. The origins of the effects cannot be assigned with certainty but the observed changes are consistent with a mechanism involving strain from the molecular folds on the crystal surfaces. Corresponding to the changes in dimensions are significant changes in unit cell density and apparent changes in degree of crystallinity. The effects of cell variation no doubt play a role in the variation of many macroscopic physical properties with physical history of commercial polymer samples.

Stacking Faults in Alloys.—One basic parameter that determines the mechanical properties of materials is the stacking fault energy. Since technologically useful structural materials generally consist of combinations of several different phases having different chemical compositions, it is desirable to determine the effects of phase and composition changes on the stacking fault energy. The electron microscope has been applied to a study of cubic and hexagonal phases in several silver-tin alloys in order to establish the effect of phase and composition changes in a simple system. Results indicate a near equality in the stacking fault energy for these two related phases.

Quantitative Microscopy. An optical photographic scanner has been constructed to evaluate photomicrographs taken from a wide range of metallic and nonmetallic materials. Measurements characterizing the internal grain and phase structures are of great

importance in controlling the quality and uniformity of materials. Machine-evaluated photographs are being compared among different laboratories for the purposes of improving capability.

Adherence of Porcelain Enamel on Aluminum.—The Porcelain Enamel Institute is sponsoring a Research Associate Program to investigate the adherence of porcelain enamel to aluminum. The electron microscope and electron probe microanalyzer have been applied to study the interface between the two materials. Some basic differences near the interface have been observed between porcelain enamel-aluminum alloy systems showing good adherence with subsequent spalling resistance and those combinations which fail. The electron microscope shows a layer of foreign material present at the interface of those systems which have poor adherence. The electron microprobe also detects an enrichment of magnesium at the interface region on certain alloys that exhibit poor coating adherence. Neither the layer nor magnesium buildup was observed on the systems having good adherence.



Tapered-section through a porcelain enamel-aluminum alloy specimen as seen by the electron microscope shows (arrow) the presence of additional material at the interface. This material affects the degree of adhesion coating.

DATA ON THE PROPERTIES OF MATERIALS

This program area includes making precise measurements of materials properties and the development of new measurement techniques. It also includes fundamental investigations of physical phenomena of importance to science and industry (corrosion, fatigue and fracture, etc.). Establishing the laws relating the physical properties of materials to their characteristics is of prime concern. Theoretical work plays a major role in this area.

Mechanical Properties and Strength of Materials

Elastic Constants of Lower-Symmetry Tetragonal Crystals.—A complete analysis of the elastic constants of CaMoO_4 were made in order to predict mechanical stability in applications such as a host crystal for laser rods.

This analysis was also of scientific interest. Accurate determinations of elastic constants of single crystals are based on resonance frequency or wave velocity measurements. Wave velocity measurements can be made upon small specimens and as a function of pressure; however, the analysis is complicated for low-symmetry crystals and some of the elastic constants depend upon choosing the appropriate root of an algebraic equation. Fortunately, the elastic constants appear linearly in the expressions associated with the resonance technique; therefore, no ambiguity arises. In this experiment the complete analysis extended the elastic constant determination usually effective only for high-symmetry tetragonal single crystals to lower symmetry tetragonal single crystals.

Properties of Electrodeposited Copper.—An IMR Industrial Research Associate, sponsored jointly by the American Electroplaters' Society, Inc., The International Copper Research Association, and the Copper Development Association, has obtained comprehensive data on the properties of electrodeposited copper. Types of plating baths used in the program included sulfate, cyanide, pyrophosphate, fluoroborate, and amine. A total of about 150 sets of operating conditions were used, comprising variations in bath composition, temperature, and current density. Properties measured and correlated include tensile and yield strengths, modulus of elasticity, elongation, internal stress, fatigue limits, hardness, density, electrical resistivity, thermal expansivity, structure, and chemical composition. Effects of cold working and annealing, and effects of cryogenic and elevated temperatures on some of the above properties were also determined.

Ultra Stable Maraging Steel.—Measurements over a two-year period of the temporal dimensional stability of a 12 percent Nickel maraging steel heat treated to meet requirements for a gage block material

indicate that it is one of the most stable materials ever observed in the continuing NBS gage block program. The long time dimensional stability of this important new class of steels has never before been reported. Stability of the two test blocks were $-0.03 \mu\text{in/in/yr}$ and $+0.10 \mu\text{in/in/yr}$ respectively.

Studies of Fiber Reinforced Metals.—The problem of identifying the constituents as well as their distribution in an aluminum-silicon alloy containing sapphire fibers was solved, in part, with the use of color photomicrography and the microprobe analyzer. Factors contributing to the strengthening of vapor-deposited copper reinforced with tungsten wires were correlated with ductility, fracture types, and resistivity measurements.

Microplasticity in Metals.—The accuracy of microstrain measurements has been improved by two developments. An IMR designed specimen enables measurements to be made over a well defined gage length, and precision measurements of temperature of the specimen allow for the correction of temperature effects caused by loading and unloading. Neither of these features have been indicated in reports emanating from other laboratories.

Fatigue Cracking in Chromium Coated Iron.—Chromium diffusion coatings on commercial Armco Iron lead to changes in microstructure, stress distribution, and strength in the outer layer of the base material. These factors affect the mechanism of fatigue formation. In coated samples, the crack initiates below the Cr-rich zone, generally in the grain boundaries where precipitated chromium carbides were discovered. Residual tensile stresses introduced below the coated layer by the process are considered an accelerating factor for crack formation.

Fatigue Crack Propagation Under Constant Load Amplitude.—A theoretical-mathematical evaluation of fatigue crack propagation in cyclic bending was completed. It was found that fatigue crack propagation can be expressed as a function of stress intensity factor. The experimental results agree closely with the theoretical model. The influence of localized (heterogenous) structural changes on the proposed model were also investigated. It appears that the length of fatigue crack propagation and the rate of propagation cannot always be expressed as a function of plastic zone size. This was related to the anisotropic conditions of plastic deformation.

Fatigue Due to Reversed Bending and Reversed Torsional Loads.—A recently completed investigation showed that fatigue damage due

to reversed bending loads did not reduce the resistance to fatigue crack initiation under reversed torsional loads. The experiments were conducted by stressing (bending) aluminum alloy specimens until a number of small fatigue cracks had developed, then determining the number of stress cycles required to develop small cracks under reversed torsion. It was found that this number was not significantly different from that required to give equivalent damage in specimens that had not been stressed previously. The reversed sequence was also investigated and it was found that prior stressing in reversed torsion did not affect the resistance to crack initiation in reversed bending. The results indicate that in alloys of this type the damaging effect of fatigue stressing is limited to a relatively small proportion of the crystals which are subjected to potentially dangerous ranges of stress.

Stress Rupture Characteristics of a Titanium Alloy.—In contrast to some theories and some experimental observations on other metals and alloys (e.g., copper and some steels), ductility of Ti-8Al-1Mo-1V increased with increase in test temperature and decrease in strain rate in creep tests with rupture times up to 10,000 hr. Although stress-rupture properties of this alloy were greatly affected by notches at low temperature, the presence of notches of varying notch geometries had little or no effect on the behavior at elevated temperatures.

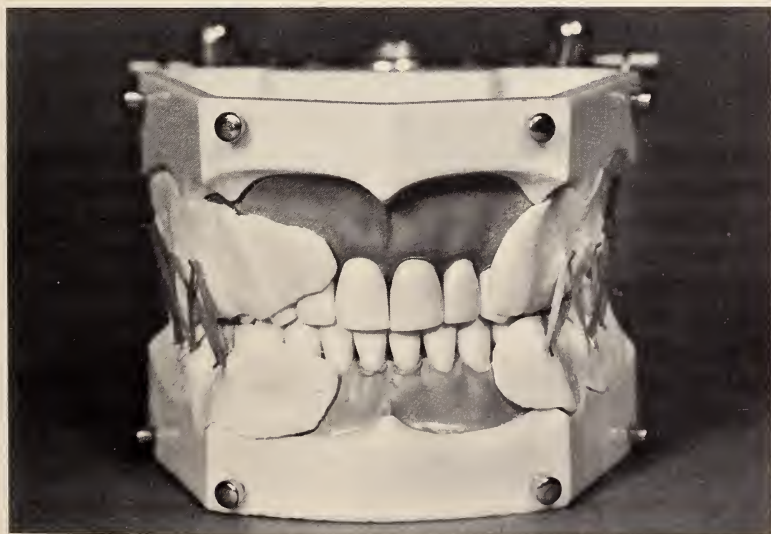
Improved Dental Cements.—Zinc oxide-eugenol (ZOE) cements have found many applications in dentistry. This puttylike material is more compatible with both the hard and soft tissues of the mouth than other dental restorative materials. Previous investigations at IMR have shown that incorporation of *o*-ethoxybenzoic acid (EBA) and certain additives to ZOE mixtures greatly improved the mechanical properties of these cements.

Recent studies have shown that aluminum oxide acts as a reinforcing agent and further enhances mechanical and physical properties. The high strength and low solubility of the Al_2O_3 reinforced materials, as well as their ease of manipulation, suggested their use for the final cementation of cast crowns and as insulating base. Cements based on the formulations developed at NBS have become commercially available and have been well accepted by the dental profession. Further modification of these products incorporating copolymers of relatively low elastic moduli are showing considerable promise as a rapidly insertable, long-duration temporary restorative. A material of this type is of special interest to the Armed Forces since the prolonged life of this material would permit delay of necessary definitive treatment.

Rapidly Fabricated Surgical Splint.—A large percentage of all injuries incurred in combat are manifested as damage to facial structures. Among these, fractures of the mandible (jaw) commonly occur. The presently employed technics of splinting fractures of the mandible using archbars are not only traumatic to the oral tissues, but are also time-consuming and require highly trained personnel and specialized laboratory equipment and facilities.

A polymeric material suitable for the rapid fabrication of splint-type appliances for the fixation and stabilization of fractures of the mandible has been developed for the Department of Defense. The doughlike product formed on the mixing of the powder and liquid can be readily adapted to the cervical third of the intact teeth. Polymerization occurs in the mouth within 4 to 7 min. at peak temperatures not exceeding 50 °C. The polymerized product exhibits sufficient strength, rigidity, and dimensional stability to function as a splint. Preformed, spring-like lingually inserted clasps prevent the buccal displacement of the polymerized splint and serve also as anchor points for interarch elastics. The use of the "rapid splint" precludes the need for cast arch-bars and complicated wiring procedures.

Limited clinical tests for this application and for the stabilization of alveolar fractures and avulsed teeth have been conducted successfully. The same polymeric material may find application as a hard denture reliner, as an orthopedic splint and for the stabilization of metal implants.



Rapidly fabricated surgical splint made from a dough-like polymer which hardens in the mouth in 4 to 7 minutes.

Reactivity and Corrosion

Reaction of Oxygen Atoms with Olefins.—Studies of the reactions of oxygen atoms with olefinic molecules at cryogenic temperatures have been completed. They have relevance to problems of atmospheric pollution. The transition state model adopted for the initial stage of the reaction is one in which the oxygen atom approaches the olefinic bond in the plane of the molecule. Further, there is a force between the oxygen and the hydrogen atoms attached to the olefinic carbons on the O atom approach side. This interaction, though weak, is sufficient to inhibit the migration of the hydrogen atom in the passage from the transition state to final products. The model has general applicability and has been successful, for example, in predicting the course of reaction for 2-methyl-2-butene.

Second Layer Migration and Adsorption Displacement.—By combining a field emission microscope with a molecular beam apparatus and operating the microscope at liquid helium temperatures, it was possible to make detailed observations of second-layer migration of hydrogen, nitrogen, and carbon monoxide on a tungsten surface. The tungsten emitter was placed with its axis perpendicular to the molecular beam. The characteristics of the second-layer migration with trapping on free sites were determined. The density of the free sites as well as surface geometry appear to be instrumental in the structuring of the sharp boundary shape as the migration progresses over the surface. An unusual effect was that of the flow of nitrogen around the 100 planes of the tungsten surface, leaving them as uncovered islands until most of the rest of the surface was completely nitrogen covered.

It has been known that carbon monoxide or nitrogen are capable of replacing hydrogen adsorbed on tungsten. With the molecular beam field emission microscope apparatus using sequential deposition and field emission observations of the surface, it was found that the displacement occurs even at 4.2 K. The displacement is not complete, however, and a relatively strong binding state of hydrogen remains undisplaced.

Electron Interaction With Chemisorbed Diatomics.—Studies of processes occurring upon electron impact on chemisorbed diatomics have led to useful information in assessing the chemisorption process. The low energy (<75 eV) electron-impact conversion of a weakly chemisorbed molecular γ species of nitrogen on tungsten to a more strongly bound chemisorbed species has been observed. This new state, designated as λ , effectively doubles the concentration of strongly bound nitrogen after the conversion from γ to λ by electron impact. Neither N_2^+ nor N^+ were detected with electron bombardment of the nitrogen

covered tungsten surface. In a mixture of $^{14}\text{N}_2$ and $^{15}\text{N}_2$ absorbed in the γ state, isotopic mixing was not observed in the residual γ state layer after electron impact.

Combination of Surface Measurement Techniques.—A technique has been devised to enable low-energy-electron diffraction (LEED) and field-electron microscopy (FEM) investigations to be made on a single specimen. Since LEED primarily determines long-range single-crystal plane surface structure and FEM detects and characterizes short-range adsorption effects, the two individual techniques complement each other. This removes some of the shortcomings of the techniques when used separately, and enables meaningful adsorption sensitivity comparisons to be made.

Microbial Corrosion of Iron.—An investigation of the anaerobic corrosion of iron has shown that sulfate-reducing bacteria cause corrosion by a cathodic depolarization mechanism, utilizing phosphate as an electron acceptor. Iron phosphite is produced as a result of this corrosion process. The currently accepted theory is that sulfate is reduced with the production of iron sulfide.

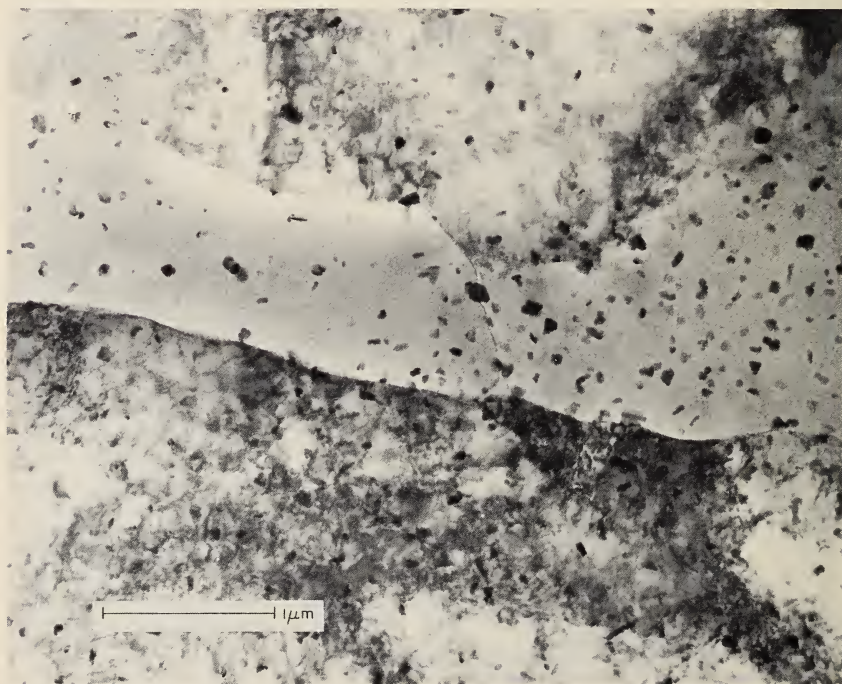
Stress Corrosion Studies.—The stress corrosion of aluminum alloy extruded plates used in the aircraft industry occurs much more readily when the plate is stressed in one direction with respect to the extrusion direction than in another direction perpendicular to this. Scanning electron microscopy revealed that crack initiation occurs in very short times without regard to the stress direction, but that crack propagation does, and this controls the time to failure.

The Reactivity of the Components of Tooth Structure.—A common cause of failure of a dental "filling" is the inadequate seal between the surfaces of the hard tooth tissue and the materials used to replace tooth structure lost through decay or accident. The need for a truly adhesive restorative that would enhance the quality of the marginal seal has been recognized for many years. Lack of information regarding the chemical and physical characteristics of the surfaces of tooth structure has retarded progress towards the development of such a material.

An investigation of the reactivities of the components of human tooth structure has as its objective the determination of those substances that will bond to or modify tooth surfaces in the aqueous environment encountered in the oral cavity. Results obtained strongly suggest that the collagenous constituent, as well as the mineral portion of tooth structure, presents a vast number of reactive sites to which the bonding of a restorative may be achieved. A knowledge of the

substances that will bond at available or newly activated reactive sites may lead to the development of a biologically and mechanically suitable adhesive dental restorative and a colorless transparent "paint-on" that could be applied to pre-carious decalcified areas to arrest tooth decay in its initial stages.

Microscopic Studies of Corrosion.—The susceptibility to stress corrosion cracking of some commercial aluminum alloys is known to depend on the direction and magnitude of the applied stress and on the temper of the material. The technique of transmission electron microscopy was applied to study directly the internal structures in alloys subjected to different tempering treatments. The more resistant material was found to contain fewer defects and larger internal precipitate particles. The observed directional corrosion susceptibility could be correlated with internal boundaries in the material but not with other structural characteristics.



An electron microscope photograph of the internal structure of an aluminum alloy subjected to stress-corrosion testing.

Adsorption of Polymers on Surfaces.—The adsorption of polymer molecules on surfaces is being investigated for both technological and scientific reasons. The nature of the interaction of these molecules with surfaces is important to the field of adhesion, coatings, and composites.

Scientifically, it is important to understand the changes in the conformation of a molecule upon its interaction with a surface and to establish the number of contacts of the long chain macromolecule with the surface. The work which is still underway has provided significant understanding of these parameters. As part of this study, the adsorption of a simple smaller molecule, stearic acid, was studied to help interpret the results obtained from polymers. Mercury was selected as a substrate in order to provide a well-defined, smooth, and clean surface. The measurements were carried out using stearic acid labeled with radioactive C-14, and the development of new measurement techniques was required. The results showed a surprisingly high concentration of stearic acid on the mercury surface, demonstrating the ready accessibility of the surface to these organic molecules.

Thermal Decomposition of Organic Aromatic Liquids.—The thermal decomposition of biphenyl, phenyl ether, and their eutectic mixture has been studied in the temperature region of 350 to 450 °C. The eutectic mixture is a well known heat transfer liquid and is to be used in many advanced type power generating systems including those designed for use in remote installations. Detailed identification of the 20 or more decomposition products and measurement of their rate of formation is being carried out. The products from the pyrolysis of biphenyl are: hydrogen, methane, benzene, 3 terphenyl isomers, and 5 quaterphenyl isomers. The eutectic mixture yields similar products in addition to phenol and phenoxy ether type derivatives. Quantitative analyses are performed by vapor phase chromatography of the liquid and solid products. From the rate data of the decomposition of initial material and formation of products at various temperatures, activation energies are calculated and compared. Data of this type are important in determining reaction mechanisms by which these fluids degrade thermally and in extrapolating the results to the conditions of technological applicability.

Thermodynamics and Kinetic Data

Model of Two-Dimensional Polymer Crystallization.—A model of two-dimensional polymer crystallization by chain folding was treated by equilibrium statistical mechanics. The Laplace transform of the partition function with respect to the length of the polymer chain was obtained in exact analytical form. The model leads to thermodynamically well-defined chain-folded crystals. Under certain circumstances in the limit of an infinitely long polymer chain, the model shows a second-order phase transition from an "extended-chain" crystal to the chain-folded crystal.

Theoretical Chemistry.—Two physically dissimilar problems which are identical from a mathematical point of view have been investigated. They are:

(1) The simplest nontrivial example of the excluded volume problem which arises in the discussion of the mean dimensions of chain polymers. In this example, the only excluded volume restriction on the overlap of units of the polymer chain which is considered is that no unit of the chain can overlap the first unit. The effect of this restriction on the mean square end-to-end distance in the chain is evaluated in the cases where the polymer chain is confined to a solution surface and where the chain molecule is in solution.

(2) The time-dependence of the momentum autocorrelation function of a heavy isotopic particle in a harmonic crystal in the case where the crystal is treated as finite. It is shown that in the case of a one-dimensional crystal the momentum autocorrelation function is approximately a simple exponential. Explicit estimates of the accuracy of this representation of the autocorrelation function are obtained and are of interest because the heavy-particle-in-a-crystal system serves as an example of a particle exhibiting Brownian motion.

Annual Index of Worldwide Published Thermochemical Studies.—As a part of its continuing program on the collection and evaluation of chemical thermodynamic data, the IMR Thermochemical Data Center has participated for several years in preparing the Bulletin of Thermodynamics and Thermochemistry. This annual Bulletin, under the auspices of the International Union of Pure and Applied Chemistry Commission on Thermodynamics and Thermochemistry, contains listings of the thermodynamic work in progress in laboratories throughout the world and an index to the published thermodynamic literature of the past year.

The National Bureau of Standards makes its principal scientific contribution to the Bulletin in the preparation of the index. Its continuing program in the collection and critical evaluation of thermodynamic data provides a natural resource for this information as a step in a more complete study which is carried out by the NBS Thermochemical Data Center. The Bureau contribution covers the general field of inorganic and metal-organic compounds and indicates properties that were measured for each compound listed. Organic compounds are covered by another group. The current issue of the Bulletin includes over 2,300 references, with about 8,000 citations to chemical species in the index.

In the preparation of the Bulletin this year, all material was originally entered on punched tape. Computer programs and techniques were then used for editing, sorting, arranging, and final output

of camera-ready copy. This serves as a pilot project for the eventual mechanization of data handling and storage for the general files of thermodynamic data material by the Center.

Thermochemistry of Fluorine—Containing Oxidizers.—Several fluorine containing compounds have important potential use as storable liquid oxidizers for special rocket propulsion problems. A study of the heat of formation of chlorine trifluoride has resolved suspicions that previous data were in error by several kilocalories per mole, and has provided a more accurate value for the heat of formation. The extremely reactive gas was reacted with excess hydrogen and a liquid water phase in a constant-pressure flame calorimeter, giving a mixture of dissolved hydrofluoric and hydrochloric acids. The flame calorimetry technique has demonstrated its adaptability to a number of problems of this kind with increasing accuracy and is being extended to chlorine pentafluoride and to trifluoramine oxide.

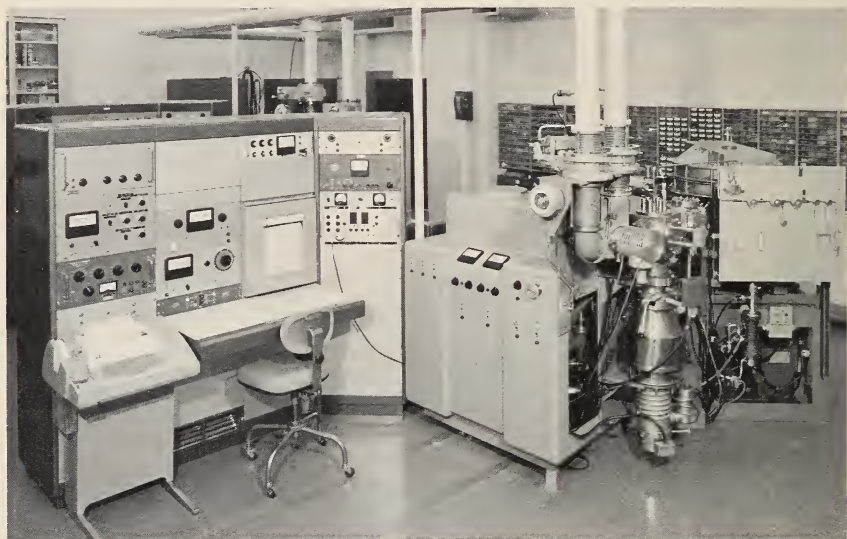
Chemical Kinetics Mass Spectrometry.—A major objective of the chemical kinetics mass spectrometry program has been the measurement of rate constants for the reactions of atoms with organic compounds. The first series of measurements involved reactions of atomic nitrogen with various olefins and acetylene. This work has now been extended to reactions of atomic oxygen. Results have been obtained for a series of alkanes and alkyl halides from about 200 to 650 K.

Cracking of Hydrocarbons.—Decomposition of hydrocarbons at high temperatures has been studied by millisecond heating with a shock wave. In the initial step a carbon-carbon bond is broken, forming two free radicals which may later react or further decompose. Detailed studies have shown which bonds break, how rapidly this occurs, and the relative importance of each route.

Potential Energy Surfaces for Chemical Reactions.—Calculation of rates of chemical reaction and reactive cross sections requires exact quantum mechanical calculation of the forces acting between molecules. The contours of this "potential energy diagram" were calculated for the HHD system. This is the first of several such surfaces which are being calculated as input to theoretical determination of reactive cross sections for elementary processes. Calculations of such "exact" surfaces, i.e., ones for which no approximations are made in the physics of the system, are becoming competitive in cost with the experimental rate determinations.

Photoionization Mass Spectrometry.—The Mark I Photoionization Mass Spectrometer utilizing a photon energy bandwidth of 1 Å at any desired wavelength throughout the vacuum ultraviolet region (1500

to 600 Å) has provided values of highest known precision and accuracy for threshold measurements and thermodynamic properties for a number of gaseous molecules of importance to the understanding of physical and chemical phenomena in terrestrial and extra-terrestrial atmospheres. Also, the past year has seen the completion and initial operation of the Mark II Photoionization Mass Spectrometer with optical and ion detection capabilities beyond those of the Mark I machine. This new and powerful tool was constructed for the study of "ion spectroscopy"; i.e., the detailed investigation of excitation processes of ions including the relationship of various spectroscopic phenomena to the modes and kinetics of ion formation and decomposition. Preliminary experiments have already shown the feasibility of achieving significant advances toward these goals and toward still further improvement in the determination of thermodynamic properties of molecules and ions.



Mark II Photoionization Mass Spectrometer developed for the detailed investigation of excitation processes of ions.

Vacuum Ultraviolet Fluorescence Studies.—Upon irradiation in the vacuum ultraviolet a molecule may dissociate and produce an electronically excited fragment which fluoresces. The threshold energy of the incident light needed to produce the fluorescence should give an upper bound for the dissociation energy of a molecule. This method has been successfully applied to a number of cyanogen compounds and the heat of formation of CN has been derived. Photodissociation processes to form electronically excited fragments have been correlated

with the absorption processes and reactivities of electronically excited fragments have been measured from studies of quenching of the fluorescence.

Photolysis of Simple Molecules.—The nature of the particles into which an excited molecule fragments has been investigated for such molecules as CH_4 and CH_2CO . A twin cell technique was used in which two reaction systems are simultaneously but independently irradiated. The photochemistry in the individual cells may then be directly compared with respect to quantum yields and modes of fragmentation.

Superconducting Metal Compounds.—Studies are now in progress to determine the constitution and degree of atomic ordering in alloys containing metals of the platinum family. As a result of these studies, several metal compounds have been discovered to lose their resistance to the flow of an electrical current (superconductivity) at extremely low temperatures. The new superconductors are $\text{Cr}_{72}\text{Os}_{28}$, Cr_3Ir , Cr_3Rh , and Nb_3Os —all having the Al $\bar{5}$ or beta-tungsten type crystal structure. The temperature at which these compounds become superconducting is found to be dependent on the degree to which each atom type tends to segregate in occupying the available atomic positions. A high transition temperature is desirable for many practical applications.

Solubilities of Calcium Phosphates.—Measurements on the solubilities of CaHPO_4 , $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$, $\beta\text{-Ca}_3(\text{PO}_4)_2$, and $\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6$ have been carried out in the ternary system $\text{Ca}(\text{OH})_2\text{-H}_3\text{PO}_4\text{-H}_2\text{O}$ at various temperatures between 5 and 37 °C. From these data it is possible to calculate the relative stabilities of the calcium phosphates and to predict what reactions can take place under the conditions of their occurrence. The data lend themselves to the calculation of a self-consistent set of thermodynamic quantities for this group of important inorganic compounds, which occur in hard tissues such as tooth and bone and in many soft tissues. They also have considerable industrial importance as fertilizers, in sugar purification, in sewage treatment, as minerals, and in fluorescent lamps. In all these connections, the solubility characteristics of the various calcium phosphates determine the biochemical behavior or the conditions of manufacture or use.

Vaporization of High Molecular Weight Organic Liquids.—The rates of molecular vaporization of large organic molecules have been measured. These molecules have molecular weights ranging from 500 to 2000 and are of the type used to plasticize polymeric materials. Hence their ability to evaporate has a considerable technological importance. The results of the study demonstrate that the energy of

evaporation can be relatively small since these flexible molecules apparently coil up and evaporate approximately as spheres. An understanding of these processes has an application not only to the plasticization phenomenon but also to space and environment related questions as well as to the thermal decomposition of high polymers.

Reference Materials for Low Vapor Pressure Measurements.—Five reference materials are being developed for calibration and checking of equipment and techniques used in measuring vapor pressures in the 10^{-1} to 10^{-6} torr (1 torr = 133.32 N/m^2) range. These high purity samples are cadmium, gold, silver, platinum, and tungsten. The materials will be suitable for a variety of pressure measurement methods and will cover the temperature range 500 to 3000 K. The materials will be particularly useful in the fields of high temperature chemistry and refractory metals. Thirty laboratories from the United States, West Germany, and Italy are cooperating in the program.

Equation of State in the Critical Region.—Studies around the critical point in the gas-liquid region of a one-component system and in the liquid-liquid phase separation of a two-component system continue to be of interest to test recently proposed theories. Accurate measurements on a liquid-liquid system are in excellent agreement with those on one-component systems, indicating that a common basis underlies the equations of state in the critical region of quite different systems.

Melting Point of Aluminum Oxide.—The melting point of aluminum oxide (Al_2O_3) has been determined in controlled environments in an attempt to explain the variance in seemingly reliable values reported in the literature. Employing a modified quench technique, melting point experiments have been conducted in specially designed induction furnaces capable of heating small samples under blackbody conditions to 3000 °C in vacuum, 2600 °C in helium or argon, and 2400 °C in air. The data indicate that the melting point of Al_2O_3 in vacuum is 2051 °C (IPTS). Maximum overall uncertainty is estimated to be ± 6 °C. Precision of the measurements is ± 1.5 °C.

Although the alumina point has been suggested as a secondary temperature reference point, the investigation showed that environment has a significant effect on the melting temperature. Vacuum conditions are believed to give the most reliable indication of the melting point of Al_2O_3 single crystal specimens (sapphire). On the other hand, each of the various atmospheres, including air, yielded significantly lower melting points. To fully resolve the problems related to this material and other potentially useful secondary temperature standards, an international task force has been formed under the auspices of the Commission on High Temperatures and Refractories, International



Single crystal test specimens (sapphire) utilized in the precise determination of the melting point of alumina (Al_2O_3). The specimen on the left exhibits characteristics melting upon heating at 2052 °C; the specimen on the right remains unmelted after treatment at 2050 °C.

Union of Pure and Applied Chemistry. The task force, chaired by an NBS staff member, consists of 13 scientists representing nine countries.

Monte-Carlo Studies of Self-Interacting Polymer Molecules.—Analytical theories of the configurations of polymer molecules cannot treat in an exact manner the mutual interference and attraction of different parts of the molecule with each other. In order to investigate these effects, polymer molecules were simulated by chain lattices. An attractive energy was found for each lattice for which the two effects compensate each other. Also some of the parameters characterizing the configurations were found to be independent of the lattice model that was used.

Diffusion in Metals.—The relation between two major types of diffusion measurements has been determined with greatly improved precision. From calculations based on the theory of atomic diffusion in metals, a relationship between (1) tracer diffusion experiments in homogeneous alloys and (2) interdiffusion experiments in nonhomogeneous alloys was proposed. This relation has been confirmed by experiments in AgCd alloys.

Interdiffusion often is important in practical applications but is difficult to measure accurately by direct means. The present work shows in detail how to use tracer measurements, which are very accurate, to predict interdiffusion results. This work also has wide im-

plications for other diffusion applications, since it demonstrates the importance of several previously neglected terms in the thermodynamic diffusion equations.

Heat Capacity of Material in Glassy, Crystalline, and Liquid States.—The heat capacity of any glassy material has usually been assumed to be similar to, though uniformly slightly higher than, that of the same material in the crystalline state. A more careful examination of this assumption has been made by measuring and comparing precise values for the heat capacities of the model material diethyl phthalate in the glassy, crystalline and liquid states. Although the above assumption may be satisfactory for rough purposes, it is inadequate when a precise comparison is needed. In the temperature range from about 30 to 75 K, the heat capacities of diethyl phthalate glass are lower than those for the crystal by more than the limit of uncertainty of the measurements. Examination of existing literature shows that this is also suggested for some half dozen other materials over discreet temperature ranges. The results for diethyl phthalate also bear out recent indications that at the lowest temperatures investigated, around 10 K, the heat capacity of a glass is much higher than that of the crystalline state of the same material. This fact may be connected with aspects of the molecular structure of glasses which also manifest themselves in other types of behavior such as dielectric relaxation, ultrasonic absorption, nuclear spin relaxation and thermal conductivity. The diethyl phthalate data also show unequivocally for the first time that the heat capacities for the annealed glass and for the quenched glass differ by more than the uncertainty of the measurements.

Actinometry.—In order to unravel the mechanism of chemical events induced in matter by interaction with high energy radiation, it is important to determine the relative importance of ionization of the molecules versus the raising of the molecules to highly excited states. Using as radiation source an argon resonance lamp which emits photons of 11.6 to 11.8 eV energy, saturation ion currents induced in various organic compounds were compared with saturation ion currents in a compound having a well known quantum yield of ionization. In this way ionization quantum yields for these compounds at the argon resonance lines were accurately determined.

The photolysis of several hydrocarbons at the argon resonance lines was studied. The modes of decomposition of the molecules excited to neutral levels above the ionization energy as well as the ionic mechanisms occurring in the high energy photolysis were elucidated. The measurement of saturation ion currents permitted the accurate determination of the quantum yields of the various product molecules.

Point Defects in the Alkaline-Earth Halides.—Based on current theories of the properties of highly ionic crystals, methods have been developed for semi-empirical calculations of the formation enthalpies of vacancies and interstitials in the fluorite structure, and applied to CaF_2 , SrF_2 , and BaF_2 . This represents the first successful excursion of this kind of theory beyond the alkali halides. The calculated values successfully account for the predominance of anion Frenkel pairs in these substances, and give formation enthalpies in close agreement with measured values. They also account for the measured enthalpy of solution of NaF in CaF_2 . One of the interesting results of these calculations is to suggest that in CaF_2 containing trivalent cations, cation vacancies may exist in addition to anion interstitials. The latter are generally accepted as the charge compensation species, but experimental research in the literature occasionally hints at the presence of cation vacancies as well.

Electric, Magnetic, and Optical Properties

Dielectric Properties of Foliage.—The dielectric properties of foliage have been measured in a coaxial cylindrical sample holder at frequencies from 10^5 to 4×10^9 Hz. Line admittances were determined using transformer bridges below 2.5×10^8 Hz and a slotted line above 2.5×10^8 Hz. Values of both the real and imaginary parts of the dielectric constant were found to approach those of water at the highest frequencies and to approach very high values ($\approx 10^4$) at the lower frequencies. The data are of value to researchers interested in the effects of foliage on electromagnetic radiation, and the method is generally applicable to a wide variety of biological materials. This data was needed by the Navy to solve communications problems in Vietnam.

Photoelastic Constants of Glasses and Single Crystals.—Techniques using a combination of hydrostatic pressure and uniaxial loading have been used for the determination of the photoelastic constants of glasses and single crystals. This combination is unique with the Institute for Materials Research and represents a new development in this area. The symmetry of a crystal determines the number of photoelastic constants, and the number of directions in which the crystal must be stressed to evaluate all of the constants. Experimentally, the problem resolves itself into determining the absolute changes in refractive index and the birefringence induced by stress. In the past, determination of the absolute changes in refractive index have proved to be most difficult. The use of hydrostatic stressing offers several advantages: (1) the precision of measurement is much greater, (2) the dependence of refractive index upon density may be determined directly, (3) much higher stresses may be obtained, (4) loading is

uniform, and (5) the shape and orientation of the cut specimen becomes much less critical.

A knowledge of the photoelastic constants of single crystals and glasses is of great interest because of optical distortion induced in laser rods by thermal stresses.

Thermo-Optical Studies on Glass.—A study of oxide glasses of different types—silicates, borates, calcium aluminate, germanates, and phosphates—is now being made to determine the effect of composition on the temperature coefficient of refractive index. An interferometric method has been used for the measurements. All of the specimens are found to have positive coefficients of refractive index except for the borate glass, which is slightly negative up to about 400° C.

By combining data on the change of refractive index as a function of temperature with data on its change with density, it is possible to separate effects on polarizability caused by (1) the change in temperature alone, and (2) the associated change in volume. It is found that the temperature term is the more important one for the glasses studied thus far. This is in contrast to the behavior of crystals, where the volume term is of greater magnitude. The change in refractive index of optical materials with temperature is of great importance to designers of optical systems that operate over wide temperature ranges.

Transverse Electroreflectance Using a New Modulation Technique.—A new measurement technique has been developed which allows a detailed study of the electronic band structure of semi-insulating materials. In this method the sample is modulated by means of a large electric field (10 to 100 kV/cm); the field is applied between two evaporated gold electrodes on the front face of the sample as an a-c square wave of frequency 1 to 10 kHz. The technique has been used on a number of semi-conductors and insulators in collaboration with Brown University. Because of the square wave form, one has a better knowledge of the magnitude of the applied field. Hence it is possible to study quantitatively the change in position, magnitude and shape of the reflectivity peaks and edges as a function of the field. Another advantage is the opportunity to explore the polarization dependence of the absorbed or reflected light. A wide variety of materials have been studied. Excitons in CdS were investigated and similar measurements were performed on ZnS, ZnTe, Si, GaAs, Mg₂Si, Mg₂Ge, and Mg₂Sn.

Development of Unique NMR/NQR Equipment.—The design and construction of an NMR/NQR (nuclear magnetic resonance/nuclear quadrupole resonance) spectrometer has recently been completed. It is being used for NMR and NQR line shape studies, and for studies

of a variety of dynamic processes such as molecular diffusion, molecular rotation, and chemical exchange processes. The spectrometer, which can be used to study dynamic processes which occur in the time interval 10^{-5} to about 10^{+3} seconds, has several unique features. Its high power (15 kW) and fast recovery time (about 4 μ s) make it possible to study dynamics of solids as well as liquids, and also very slow motions in viscous systems (such as polymers) via the rotating reference frame technique. The unit has a continuously variable frequency range of 2 to 28 MHz, which is necessary for pure quadrupole studies, and its single coil design permits low (below 4K) and high (above 1200K) temperature studies. A second unit has been built to study NQR line shapes and intensities. The intent of the work in this instance is to develop methods for accurate temperature measurements below 1K.

Analysis of Magnetic Resonance Data.—A major standards experiment concerned with improving the accuracy and interpretation of data obtained in magnetic resonance experiments has been completed. Measurements were made of ratios of electronic to nuclear g -factors which will make it possible to determine g -factors to an accuracy of one part in a hundred million. In the analysis of resonance lines containing arbitrary mixtures of absorption and dispersion, a simple correlation has been shown to exist between the degree of admixture and a system phase or frequency shift, thus enabling one to obtain the pure lineshapes by a simple and known adjustment of the spectrometer. This result is of considerable practical importance in NMR studies of metals. In related work, lineshape distortions due to large resonance signals and the concomitant errors in the observed second moments have been analyzed. A simple line sharpening device is currently being constructed, which should be capable of significantly improving resolution in magnetic resonance spectroscopy without the appreciable losses in sensitivity inherent in other methods. Finally, several novel techniques are now being developed which will make possible a *direct* determination, to a high degree of accuracy, of the absolute number of nuclear and electron spins which give rise to magnetic resonance lines. These methods will eliminate the many serious drawbacks encountered in spin concentration measurements which are made relative to standard reference materials.

Ultra Low Frequency Impedance Bridge.—Two models of a novel ultra low frequency impedance bridge were completed and put into use. These bridges make complex impedance measurements (magnitude and phase) at frequencies from 10^{-3} Hz to 10^4 Hz with an accuracy of better than 0.1 percent in magnitude and 10^{-5} rad in phase. Impedances ranging from pure capacitive to pure resistive are easily and quickly measurable. Key features of this new bridge are: (1) the use of an

operational amplifier rather than the ordinary passive impedance ratio arms to provide two voltages equal in magnitude and 180 deg out of phase to the standard and unknown arms of the bridge respectively, (2) the use of a high impedance detector, and (3) the application of a quadrature voltage to a capacitor to balance the conductance of the unknown.

Fundamental NMR Studies of Inorganic Hydrides.—Recent broad-line and pulsed nuclear magnetic resonance (NMR) studies of $\text{H}^{11}\text{B}(\text{OC}^2\text{H}_3)_2$ and $\text{HCo}(\text{CO})_4$ at IMR in conjunction with scientists at the University of Illinois, have led to a significant new understanding of the way in which fundamental magnetic interactions of nuclei determine the shape of NMR absorption spectra and the magnitude of spin-spin and spin-lattice relaxation times. One result of this work is the demonstration, both experimentally and theoretically, that the usual Van Vleck theory, which relates the second moment of an NMR absorption spectrum to the bond distances between the interacting magnetic nuclei, does not always apply if some of the nuclei have spin quantum numbers greater than one-half.

A nucleus with a spin greater than $\frac{1}{2}$ can have a quadrupole moment. If the nuclear quadrupole coupling constant is large or comparable to the NMR frequency, this quadrupole interaction must be considered. This discovery has led to the reinterpretation of some NMR structural studies with the result that some metal-hydrogen bond distances are 0.1 to 0.2 Å longer than the values reported in the literature.

The work also contributes a technique for using pulsed NMR techniques for determining quadrupole coupling constants for *liquid samples*. An experimental demonstration of this technique was given for $\text{H}^{11}\text{B}(\text{OC}^2\text{H}_3)_2$. The agreement between the pulsed NMR study of the liquid and the more difficult, but more familiar, broad-line NMR and NQR study of the solid is excellent.

Exchange-Coupled Ions in Transition-Element Complexes.—Electron spin resonance spectra of pure and doped binuclear complexes of transition metal ions have been studied to investigate the nature of the metal-metal interaction in these materials. The first observation has been made of resonance from exchange-coupled dissimilar metal ions and information has been obtained concerning the bonding in these materials. These studies are of particular interest since it now appears likely that metal-metal "bonding" in a number of inorganic and organo-metallic compounds may lead to interesting and practical electronic properties in these materials.

Ferromagnetic Beryllium Compound.—The intermetallic compound CrBe_{12} has been discovered to exhibit ferromagnetism below 50 K. Susceptibility in the paramagnetic state and magnetization in the

ferromagnetic state have been measured. Ferromagnetism as large as observed in CrBe_{12} has not been found to occur in MnBe_{12} or FeBe_{12} or in a number of other isomorphous Be compounds. Magnetic studies on these materials will add appreciably to our understanding of ferromagnetism in metals, particularly in the new class of itinerant ferromagnets, of which CrBe_{12} appears to be an example.

Soft X-Ray Spectroscopy.—Valence band emission spectroscopy serves as a useful tool in understanding the electronic structure of solids by providing information on the distribution in energy of electrons through the valence bands. In a recent study, the $M_{2,3}$ emission band of paramagnetic nickel was measured under carefully controlled vacuum and temperature conditions. Previously unreported fine structure was observed. Detailed comparison was made between the observed spectrum, available band calculations, and the related results of photoemission and ion neutralization experiments. These comparisons strongly favored interpretation in terms of the independent particle model. Marked apparent discrepancies between the soft x-ray and photoemission results, and between the L and M emission spectra, were shown to be largely accountable on the basis of different transition probability variations as a function of energy through the valence bands. In another study, a striking correlation was found between the $M_{2,3}$ spectrum of Ni in TiNi and the variation of electronic specific heat as a function of electron per atom ratio for bcc transition metal alloys.

Analysis of Ellipsometer Measurements.—The ellipsometer is an optical instrument used to measure extremely thin films. However, its use has been limited by the complicated calculations required to determine the refractive index and thickness of a film from the ellipsometer readings. For this reason, a general purpose computer program was developed to analyze ellipsometer readings. This program can calculate the properties of either a clean surface or a film on a surface, can correct for imperfect optical components or a tilt of the reflecting surface, and can determine confidence limits for the calculated values.

Polybutylisocyanate in Dilute Solution.—Measurements of the complex dielectric constant have been made on dilute solutions of fractionated polybutylisocyanate (PBIC) in CCl_4 . The measurements, which were carried out at 23 °C and over a frequency range 0.05 Hz to 200 kHz, yielded relaxation times and dipole moments of PBIC as a function of molecular weight where M_w varied from 2.5×10^4 to 13×10^6 . For low M_w ($< 250,000$) the relaxation time data indicate that the molecule is in a rigid rod conformation. As M_w increases, this rodlike conformation can not be sustained and the effects of chain

flexibility accumulate with the addition of each monomer unit to the chain. Finally at high M_w ($>10^6$) the dominant mode of relaxation is a long-range Rouse-Zimm normal mode and the conformation of the molecule is random coil. In both limits, for rigid rod and random coil, the molecular dimensions and dipole moments were calculated.

High Accuracy 2-Fluid Dielectric Measurements.—Dielectric measurements were made on a series of solid polymer specimens using newly developed capacitance cells and a 2-fluid method. This method involves making four capacitance measurements—with the sample in the cell and with sample out in each of two fluids. The advantage of the method is that the average sample thickness (a major source of error when using older conventional methods) need not be measured independently. An increase of roughly an order of magnitude in the accuracy of the measurements has been realized and standard specimens have been prepared with dielectric constants certified to about 0.01 percent.

Pressure Effects on Dielectric Properties.—The investigation of the dielectric properties of materials has been extended to include the effects of pressure. Complex dielectric constant and volume measurements have been made using newly designed and constructed equipment on both solid and liquid samples over a wide temperature range at pressures up to 2×10^7 kg/m² (about 2000 atm.). The liquid measurements provide data on the density and temperature dependence of the polarization. The solid measurements have been used to study the pressure dependence of orientational relaxation parameters. In both cases the data separate the effects of changing sample volume by means of a pressure change from those due to changing sample volume with temperature.

Dielectric Constants of $PbWO_4$ and $CaWO_4$.—The class of compounds having the scheelite structure has received considerable attention as potential host crystals for laser applications. These scheelite-type crystals have been the object of numerous investigations, but the dielectric constants of $PbWO_4$ and $CaWO_4$ have not been measured heretofore. IMR scientists recently made such measurements at 24.5 °C in air on single crystals grown by the Czochralski method.

Hall Mobility in Semiconducting $SrTiO_3$.—During the last four years an intensive study of the electronic properties of $SrTiO_3$ has been in progress. As part of this program a close look has recently been taken at the temperature and donor-concentration dependence of the Hall mobility in order to obtain information on the scattering mechanism. Data on conductivity and Hall coefficient provide directly a measure of the electron mobility. Samples with carrier-concentrations varying between 3.4×10^{17} /cm³ and 5.3×10^{20} /cm³ were investigated.

These carriers were obtained either by hydrogen reduction of the strontium titanate or by doping with Nb or La. At high temperatures (300 to 1200 K) the mobility shows an exponential behavior. It appears that the theory for scattering by optical phonons in the intermediate coupling range gives reasonable quantitative agreement with the experimental results. At very low temperature ($T < 10\text{K}$), scattering by ionized impurities limits the Hall mobility. The scattering potential is strong, invalidating weak scattering approximations commonly used. Existing partial-wave treatment gives a satisfactory description of the data.

TECHNICAL ASSISTANCE TO OTHERS

IMR gives technical assistance and advice on materials to other Government agencies, and to science and industry. Arranging conferences and seminars to facilitate dissemination of new data useful in solving materials problems is one of the services provided.

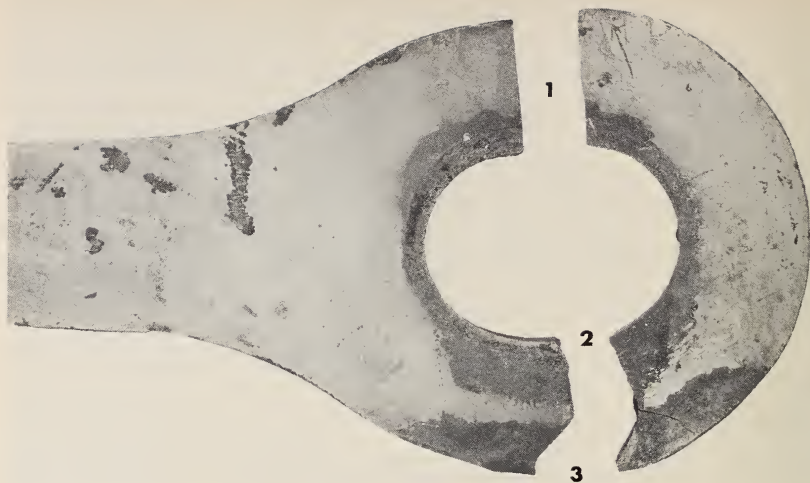
Advisory and Consulting Services

Collapsed Bridge at Point Pleasant, W. Va.—NBS is assisting in the investigation of the collapse of the highway bridge at Point Pleasant, W. Va., on December 15, 1967. The eyobar in which the initial fracture occurred has been identified and sent to NBS for examination and laboratory testing of the material, in cooperation with other interested parties.

Atmospheric Composition Studies.—NBS scientists are cooperating with the Environmental Science Services Administration in their studies of the possible variations in atmospheric oxygen content over the oceans. NBS developed a reliable method for detecting the expected small oxygen compositional differences and instructed ESSA personnel in sampling methods. Samples collected during world-wide cruises of the ESSA research ships will be analyzed in the NBS laboratories.

Life of Postage Stamp Printing Plates Increased.—Recommendations for changes in the fabrication and heat treating procedures of postage stamp printing plates have been made to the Bureau of Engraving and Printing. Early and still preliminary reports indicate that these procedures will increase the service life of the plates.

Analysis of Lung Tissue from Astronauts in Fatal Spacecraft Fire.—IMR determined the trace contaminants in lung tissue taken from astronauts killed in the fatal spacecraft fire,



On December 15, 1967, this structural member on the bridge at Point Pleasant, W. Va., failed, sending 46 people to their death and injuring many others. The member, called an eye bar, was sent to NBS for analysis to determine the type and cause of failure. Study has shown that initial brittle fracture occurred at 1; ductile fracture then occurred at 2 and 3. The peeling of the paint shows extensive deformation at 2 and 3 with a zone of almost no stress (where the paint remains) running in between. Results of this study should provide important information on the cause of the tragedy and give indications of methods needed to insure the safety of similar bridges.

Naming of Synthetic Fibers.—IMR advised the Federal Trade Commission on naming of manmade fibers according to the Textile Labeling Act. This keeps the consumer from being overwhelmed by a confusing array of names.

Conferences and Symposia

Second Annual IMR Symposium.—"Molecular Dynamics and Structure of Solids," the second annual symposium of the Institute for Materials Research, was held October 16–19, 1967 in the NBS laboratories at Gaithersburg, Md., and was attended by 250 university, Government, and industrial scientists from both the United States and abroad. The goal of the symposium was the encouragement of interdisciplinary materials measurement science and technology. Topics and speakers were selected so as to demonstrate the correlation of various techniques for the study of molecular dynamics and structure of molecular solids. The Proceedings are being published.

Symposium on Mass Transport in Oxides.—IMR and the Advanced Research Projects Agency of the Department of Defense joined in sponsoring this symposium to assess the concepts, theories, and experimental techniques currently applied in studies of mass transport in

oxides. Thirty papers were presented covering preparation of research specimens, characterization and energetics of defects, lattice dynamics, diffusion, and ionic conductivity. Panel discussions explored the need for fundamental understanding in solving technological problems in mass transport, the improvement of accuracy in diffusion measurements, and the usefulness and selection of standard reference materials. The published Proceedings of the Symposium are available.

Symposium on Mechanical and Thermal Properties of Ceramics.—Jointly sponsored by the American Ceramic Society, the American Society for Testing and Materials, and IMR, this symposium emphasized the dependence of ceramic properties upon microstructure and composition, and the importance of controlling these features of character to insure reliability in ceramics. Intended to provide a basis for understanding property-character relationships as well as criteria for the proper selection and use of ceramic materials, papers and discussion centered on properties of importance in structural and high-temperature applications—melting points, thermal expansion, thermal conductivity, thermal radiation, elasticity, viscoelasticity, inelastic deformation, and fracture. The published Proceedings of the Symposium are expected to be available in January 1969.

Material Failures Conference—"Must They Fail".—This conference held at NBS reviewed the growing problem of service failures of materials in view of the more stringent demands of today's space age technology. It was jointly sponsored by IMR and the Washington (D.C.) chapters of the American Society for Metals and the American Society of Mechanical Engineers. Six technical speakers reviewed the state of knowledge in the field; Ralph Nader, well-known safety critic, was luncheon speaker.

Quantitative Electron Probe Microanalysis.—This seminar was held June 12 and 13, 1967, to critically examine an important area of measurement in materials science. Twenty-five participants from the United States and five foreign countries were brought together for an exchange of ideas in the refinement of quantitative measurement in this area of microanalysis.

Mass Spectrometry Workshop.—A workshop on Spark Source Mass Spectrometry was held November 13 and 14, 1967, to examine the factors that influence the precision and accuracy attainable in analytical determinations by the spark source technique. Seventy participants from Government laboratories, universities, research institutes, and industrial laboratories participated in the discussion which centered on calibration, quantitation, and standard reference materials suitable for spark source work.

INSTITUTE FOR APPLIED TECHNOLOGY

The Institute for Applied Technology (IAT) addresses the Nation's need for measurements and standards which relate to the artifacts of our society. Thus, the IAT programs are concerned with technological or "engineering" measurement and standards which deal with products, commodities, devices, processes or systems. Such measurements and standards are the language of the market place for coupling user requirements with the performance capabilities of products, processes or services, and they bring order and quantification to man's use of his technical skills.

The Institute programs are oriented to industry, to the States and regions of the country, and to all levels of Government. They bridge the interface between science and its application to society's needs, and by coupling science and technology with the daily activities of commerce, industry, and Government, they stimulate economic progress. IAT activities fall into two major categories—Technical Measurements and Standards, and Technical Innovation and Diffusion.

A substantial portion of the work done in the entire Institute program is under the classification of technical assistance to other parts of the Bureau, other Government agencies, and to industry. The work in motor vehicle safety research and technical analysis is virtually all done for others. In other fields, a lesser but still substantial volume of IAT work is done at the request of others.

TECHNOLOGICAL MEASUREMENTS AND STANDARDS

This category covers the Institute effort for the broader extension of the national measurement system into the engineering and technological fields. It is a program to apply principles of good measurement, rather well defined in the science area, to the determination of performance or other significant characteristics of products, systems and devices important either in commerce or to Government needs. In essence, the objective is to find ways by which standards can be developed for items, systems, and devices which are not readily evaluated in terms of existing measurement methods.

The performance concept introduces man into the measurements and standards process, and the whole idea centers on the belief that products, processes, services, and systems can be described and their performance can be measured in terms of user's requirements without regard to their physical characteristics, design, or method of their

creation. Key to this method is the identification of measureable performance criteria for meeting the user's requirements, thereby coupling subjectively derived needs with objective technical measurement methods.

As the complexity and technical sophistication of articles of commerce increase, it becomes increasingly difficult to define those characteristics of a product which best measure its performance. The virtue of the performance concept in measurement and standards development is that it stresses the end to be attained, and leaves the means to obtain the desired end wide open.

Today many complex products are actually complete "systems", and the only feasible way to evaluate them is in terms of their performance against criteria established to meet the user's requirements. The concept encourages innovation when producers can meet the performance required by whatever design of product will do the job.

The idea of performance criteria also has ramifications beyond those concerned with articles of commerce. Our national welfare depends critically on making informed decisions related to complex social and economic policies and programs. To do this, one needs to define criteria of performance for these policies and programs and be able to measure or predict possible benefits and costs of alternative actions. Through the techniques of systems analysis and operations research, technology has provided a means for helping such decision-making.

Institute activities in technological measurements and standards range from the development of performance criteria and test methodology for individual items to the study and analysis of complex systems such as a multi-State transportation network.

Program elements include seven areas:

1. Building Technology
2. Automatic Data Processing
3. Electronic Technology
4. Systems Analysis
5. Motor Vehicle Safety
6. Engineering Materials
7. Industrial and Consumer Products

Building Technology

Buildings represent a large portion of the existing tangible wealth of this country. Annual building production continues in the range of \$100 billion, with an effect on every village, town, or city. And we are still faced with the need to produce in the next 30 years or so as many buildings as we now have, with an industry which has never really entered the mass-production stage and is still heavily dependent on hand craftsmen techniques. Spiraling construction costs have ac-

celerated the move to the industrialization of the building process, although these costs are only part of the total, which is heavily influenced by the cost of land and by tax, financial, and other social burdens. Nonetheless, the changes taking place are already generating pressure for advanced techniques for better measurements and updated standards. There is a greater need for standardization of test procedures and innovation needs to be encouraged by implementing the use of the performance concept for evaluating complex components. During the past year the IAT Building Technology program has accelerated efforts in this direction.

Activity has centered on improving the normal building code, standard and specification process by continued development of the technological base and measurement procedures needed for the process to function. Work on new methodologies has been initiated to bring about changes in the process whereby innovation in building systems and components will be encouraged. As an example, a study was initiated to determine how a broader use of performance based standards might open the way for the introduction of cost-reducing innovations into the design of low-cost housing. The project involved a comprehensive definition of the performance concept and the various steps that must be taken from the determination of user requirements to the issuance of performance standards.

The building technology program continued the traditional laboratory and field support provided to Federal agencies, State, and local Government groups concerned with building construction and technology. Technical consultation and advice was made available to industry, and the staff continued to work closely with the many private standards-making groups throughout the country.

Structural Performance of Multifamily Building System.—Load tests on a one-story portion of a new low-cost building system were arranged in a manner that simulated the structural response expected of a three-story unit. The laboratory tests showed that the prefabricated system did in fact produce a structure which complied with the intent of a local building code. The test section was subjected to simulated dead, live, and wind load trials. Individual components were also tested to obtain data on their behavior. Results obtained provided the performance criteria for evaluating the structural safety of this and similarly innovative building systems.

High Temperature Thermal Conductance of Soils.—It is anticipated that space nuclear power systems, on re-entry impact burial in the earth, may have a maximum steady-state temperature as high as 1700 °C. A little information is available on the thermal conductivity of soils at temperatures of 100 °C, but essentially nothing is known

about what happens above 500 °C. In order to provide the high temperature data needed in the safety evaluation of such power systems, tests were developed for thermal conductivity measurements on soils and other granular materials at hot-side temperatures near 1700 °C. Using apparatus specifically designed for the purpose, thermal conductivity values have been established for a number of natural or artificial samples representative of subsoils found on most of the earth.

Continuous HCl Analysis for Smokes.—Polyvinyl chloride is a common plastic used as a construction material. When it burns, hydrogen chloride is present in the smoke and a continuous, recording, analytical method was required for the monitoring of smoke tests. It was found that scrubbing the HCl from the smoke in a microscrubber, and analyzing it by means of a specific ion electrode, is a satisfactory method for determining the HCl present to a sensitivity of 20 ppm of HCl.

Reducing Flood Damage in Alaska.—Four thousand residential structures suffered approximately \$29 million damage in a disastrous flood in Fairbanks, Alaska, in August 1967. The NBS Building Research Division was asked by the United States Senate Public Works Committee to assist the stricken residents. An NBS team consisting of a mechanical engineer and a materials technologist was sent to Fairbanks in the company of Senator Ernest Gruening and staff members of the Public Works Committee. Recommendations were made to facilitate drying of homes for early rehabilitation with a minimum removal of water soaked materials. Based on an assessment in May 1968, the services provided by the National Bureau of Standards resulted in an estimated reduction of flood losses in the amount of \$5 million.

Effects of Air Pollution on Building Materials.—The presence of sulfur dioxide and ozone contaminants in urban atmospheres affects both people and materials. As part of the task of estimating economic losses caused by these contaminants, their effect on the chemical and physical properties of paints, plastics, and asphalts is being evaluated. Through laboratory tests, samples have been subjected to a simulated natural exposure of light and humidity along with controlled amounts of sulfur dioxide and ozone. Measurements were then made of the changes in color, tensile strength, brittleness, and chemical structure induced by the tests. Short term tests show that vinyl plastics absorb sulfur dioxide and show a loss of strength. The adsorption is dependent on humidity and light. As they develop, the studies should increase our knowledge of the interrelationship of the effects of weather variables and air contaminants.



Scenes such as these were common after the August 1967 flood which caused \$29 million damage in Alaska. Significant reductions in losses due to flood damage were realized through services provided by an NBS team of building systems experts.

Computer Simulation of Building Fires.—The complex interactions of a building and a fire are being studied with the aid of computer simulated models. A major problem has been the development of a building description of manageable size and form suitable for use on the computer. Another problem has been how to determine air movements induced by fires. Hot gases produced by fire tend to rise and mix with other gases in a room or building and knowledge of this motion is important for the early detection of fires. To provide preliminary information on this phenomenon, Research Associates of the Factory Mutual Engineering Corporation (working at NBS with Bureau staff) ran a parallel experimental-theoretical-in-motion study in a closed model room in which a hot spot on the floor was used to simulate a fire. The theoretical prediction was obtained by a computer calculation. By highlighting the information required for a successful calculation, guides to future research were obtained.

Performance Criteria for Exterior Walls.—A comprehensive study was made of the performance characteristics of exterior walls for one- and two-family houses. Using newly developed apparatus and new test procedures, combined compressive and racking tests, and transverse strength tests were developed to determine the criteria for measuring the strength properties and associated elastic or permanent deforma-

tions of such walls. Criteria were suggested for evaluating surface flammability of the interior components of the walls. In the area of the environmental requirements, new criteria were developed which covered maximum limits for air penetration of the walls and for the temperature difference between the room air and wall surface. Many other characteristics were included in this total evaluation study: the effects of weathering on color change, chalking, erosion, peeling and cracking of surfaces, washability, etc., as well as criteria related to resistance to impact and abrasion. The investigation revealed the paucity of information available for determining the performance requirements of exterior walls.

Conference of States on Building Codes and Standards.—Staff support was given to a number of States in organizing a National Conference of States on Building Codes and Standards. Modeled after the National Conference on Weights and Measures, the new group of state representatives used NBS facilities for a first meeting in May 1968. Organization of working committees is underway for study of code requirements, standards, enforcement, etc., with the objective of obtaining more uniform standards among the States. A majority of the States do not have research facilities, and thus the NBS building technology program findings will serve in support of the States in the National Conference.

Automatic Data Processing

The NBS Center for Computer Sciences and Technology completed its second full year of operation in fiscal year 1968. Established by a Commerce Department Order in September 1965, it carries out the responsibilities of the Secretary of Commerce under Public Law 89-306 (the Brooks Bill) for broad ADP standards development, consultation, and assistance to Federal agencies and supporting research in matters relating to the use of computers in the Federal Government. This mission necessitates close interaction with ADP managers in the General Services Administration and the Bureau of the Budget, who share responsibility under the Law for improving the use of computers in Government.

The Center program covers four areas: ADP standards (both Federal and in cooperative efforts with industry in the voluntary commercial area), consultative and advisory services, research, and computer services.

ADP Standards

Federal Information Processing Standards.—On March 11, 1968, President Johnson approved the first Federal standards for ADP. The

standards are the USA Standard Code for Information Interchange, and implementation of the code on perforated tape and magnetic tape. This action opened the way for important applications which require computers to talk to each other, to send each other messages, and to consult each other's files. Additional Federal standard recommendations in process include type fonts for optical character recognition, forms layout, COBOL programming language, and data elements and their coded representation.

Voluntary Commercial Information Processing Standards.—The first international standard in the information processing field was approved by member nations of the International Standards Organization—ISO Recommendation 646—which covered Six and Seven Bit Coded Character Sets for Information Interchange. The Center continued to work with the USA Standards Institute on a wide range of information processing standards aimed at improving compatibility between and within data processing installations. Projects include standards for magnetic tapes, disc packs, punched cards, data transmission, tape labels and formats, programming languages, operating systems, remote control user procedures, data elements and codes, computer terminology, program documentation, flowchart symbols, optical character recognition, keyboards, and interfaces.

Measuring Compliance With Information Processing Standards.—Routines are being prepared to analyze and test FORTRAN and COBOL compilers. In the hardware area, measurement procedures and test equipment were developed for laboratory certification of unrecorded standard reference digital computer tape samples. Under a cooperative arrangement with the General Services Administration, a magnetic media testing and evaluation facility is being established for long term operation at the NBS Gaithersburg site.

Dissemination of Information on Standards Activities.—Through distribution of three issues of an Information Processing Standards Tech Memo, ADP circles were kept aware of the status of voluntary and Federal standards work going on at the Center.

Consultative and Advisory Services

These services have been extended to more than 30 Federal agencies with computer related problems. The subjects range from reviewing proposed specifications for computer systems; conducting systems studies; evaluating, designing and developing software and hardware; to monitoring installation and systems performance on a continuing basis.

In addition to assistance on specific problems, the Center provides general help through:

- a. Development of techniques to be used as guidelines for those conducting feasibility studies, computer selections, or computer performance evaluations. These guides result from the experience gained in the performance of consulting services to other agencies.
- b. Operation of a Technical Information Exchange which provides Government agencies access to collections of formal literature in the ADP field, publications of manufacturers and suppliers, and resumés of Government sponsored projects.
- c. Automated information storage and retrieval systems developed for three of the collections, (1) the formal literature, i.e., books, proceedings, reports, serials, and periodicals, (2) resumés of government sponsored projects, and (3) publications that are available from the manufacturers and suppliers of ADP systems and services. Also, search tools in the form of a computer-produced bibliographic citations list and KWIC (Key Word in Context), personal and corporate author, subject category, and report number indexes are prepared monthly.

Research

For many years the Bureau has conducted research in computer science fields of particular utility in Government operations. The Center has continued this basic work, and has undertaken a variety of major projects this past year.

Selective Information Extractor.—A software system was developed to provide a simple language enabling nonprogrammers to query large files and to produce formatted output. From simple file descriptions and an ordinary English request, this system generates a complete COBOL program to process the user's files and to produce the desired reports. The system was written in COBOL to achieve machine independence.

Low-level Programming Language.—A programming language resembling an assembly language in format is being developed for problems which presently can only be adequately approached in true assembly languages. This language provides a small but complete basic instruction set, including some commands not usually found in assembly languages. Flexibility is gained by using parameters to modify basic instruction types, and by permitting a very general form of operand addressing. This approach is intended to avoid the almost prohibitive intricacy and cost of assembly language programming for third generation digital computers with their multiplicity of registers,

word formats, and instruction types. The language makes no reference to hardware registers, and it is in many respects machine-independent.

Career Referral System.—Research was undertaken aimed at establishing an automated Government-wide system able to identify all eligible employees with the requisite qualifications for a vacancy anywhere in the Federal Government.

"Magic"—A Display System.—This display system is used in conjunction with an experimental computer for applications such as computer aided design of structures, electronic circuit organization and file information retrieval.



Console of "Magic II" with drawing of a circuit element on the cathode-ray tube.

Time-Share System.—A time shared user project was carried out with the Center's experimental computer MOBIDIC. The project dealt with real time remote computer control of temperature and electrical current as related to the Hall effect.

Automated Fingerprint Classification.—A procedure has been developed, under the sponsorship of the Federal Bureau of Investigation, for determining by computer whether two fingerprint impressions were made by the same finger. The procedure uses the X and Y coordinates and the individual directions of the minutiae (ridge endings and bifurcations). The identity of two impressions is established by matching the impressions in terms of the relative distances

and relative angles of the minutiae. This matching procedure promises to be useful in an automated system in which a file of minutiae data corresponding to thousands of fingerprints of known individuals is searched. Thus the identity of an unknown person may be established using only one of his fingerprints.

Computer Services

The Computer Services Division of the Center provides computer time and programming within NBS and for many other Government agencies. The present multi-access computer, a UNIVAC 1108, is operated in a batch-processing mode using a priority arrangement under which users can obtain either special service or deferred turn-around service. At the end of FY 1968, two high-volume and five medium-volume remote terminals were fully operational. Output of publication quality is provided to both microfilm and hard copy of either printed or graphical material. A small-scale programmable computer provides a full range of conversion services between magnetic tape and punched paper tape.

Data processing services were provided to over 60 organizations including 9 executive departments and 18 agencies of the Government, the Congress, state and local governments, quasi-government organizations and universities. Programming and consultative services are also provided in support of NBS technical activities. This effort is largely in assisting NBS scientists with problem formulation and in writing FORTRAN programs.

Electronic Technology

The electronics industry is among the fastest growing and most heavily research-oriented in this country. In the context of R&D funds spent within the industry, NBS activities are relatively small. Yet the Bureau contribution has a broad pervasive influence in the field. Emphasis is placed on the characterization of basic building blocks, that is, acquiring the "nuts and bolts" knowledge needed for advances in the technology. An example of this kind of activity is the effort given to studies of the yield, performance, and reliability of discrete and integrated semiconductor devices. The NBS Electronics Instrumentation group, working with materials such as germanium and silicon, develops measurement techniques and test procedures for information on resistivity and lifetime, detection and identification of flaws, control of material properties in processing, and correlation of the properties of devices with materials characteristics. In the past year there has also been an expansion in participation with national and international standards groups.

Characterization of Germanium for Nuclear Radiation Detectors.—Semiconductor nuclear radiation detectors have been extensively used

for about eight years. Uncontrolled variations in the quality of germanium intended for this application have occurred during this period for reasons which are not well understood. Crystals are presently selected on the basis of room temperature resistivity, photoconductive decay lifetime, and etch pit density, but use of these parameters does not always permit discrimination between material suitable for detector fabrication and material which is not.

A comprehensive program is underway with the objective of developing methods for the early identification of material suitable for fabrication into lithium-compensated detectors. The program involves material evaluation, device fabrication, and device evaluation utilizing both "good" and "bad" material. Material properties being determined include routinely measured properties such as room temperature resistivity, photoconductive decay lifetime, etch pit density, and other less commonly measured properties such as Hall mobility and drift mobility at low temperatures, infrared absorption in the $11\text{ }\mu\text{m}$ region, lithium precipitation and drift rates, x-ray topography, and composition by means of neutron activation or mass spectrographic techniques.

Fabrication of gamma-ray detectors from the material and testing of the detectors is also part of the measurement program. Presently 38 germanium crystals contributed by various domestic suppliers and national laboratories are being studied.

Because of the large number of specimens, an automatic computer-controlled system for making low temperature Hall effect measurements was built in cooperation with the NBS Computer Center. This system enables a run to be made between 20 and 300 K in about 4 hr. Apart from the study, the automatic system is used to make measurements on specimens sent from a national laboratory specifically for Hall measurements.

Standard Method for Measuring the Resistivity of Silicon Wafers.—The technical basis for a standard method of measuring the resistivity of silicon wafers was developed in cooperation with Committee F-1 of the American Society for Testing and Materials. The method is an improved version of the four-probe technique most widely used in the industry. The improvements, based on work which has been underway since 1960, permit comparative measurements to be made with a standard deviation of less than 1 percent over much of the resistivity range of commercial interest.

Some of the features of the new method can be applied to calibration procedures for use on production equipment and hence will also enable improved control at various stages in the processing of wafers. Increased emphasis on requirements for control of the processing has led to extension of the work to include measurements on epitaxial or diffused layers with a variety of surface conditions,

Systems Analysis

In any program for promoting the application of technology to complex problems of industry or Government, the use of systems analysis or operations research is almost a mandatory requirement. The NBS Technical Analysis Division seeks to develop, test, and disseminate systems analysis techniques which are applicable to public sector problems in terms of program planning, resource allocation, and program execution.

The IAT systems analysis group is the largest such organization within the civilian agencies of the Government. It serves other agencies in the solution of their specific systems analysis problems, and helps these agencies to develop their own capacity to tackle complex systems problems. It also conducts research on cost-benefit analyses for Government programs.

Application of Systems Analysis to Highway Maintenance.—Considering highway maintenance as a system composed of three levels: (1) national, (2) state, and (3) operational, a study was conducted on the feasibility of applying systems analysis or operations research methods to the solution of problems at those levels. It was determined that in general the operational level, where measures and interactions are better defined, was more amenable to the application of analytic techniques. A computerized simulation model of a typical highway maintenance operational unit was developed to provide highway maintenance management with a quantitative means of evaluating the effect of different job-scheduling and resource-allocation policies on the productivity of the unit.

Systems Analysis and Problems of the City.—The Department of Housing and Urban Development asked the NBS technical analysis group to serve as "technical coach" on a project to determine whether quick and inexpensive application of systems analysis to selected city problems is feasible. The project is under the direction of the International City Managers' Association. Three cities are involved in the test. In East Lansing, Michigan, the project involves calculating optimal fire station locations. At Poughkeepsie, New York, comparison of possible city actions related to building code compliance was initiated. Model neighborhoods were studied, using cost to owner, cost to occupant, and cost to city measures of value. In Charlotte, North Carolina, through in-depth interview techniques, user perceptions of value and distance to public service facilities were studied.

Ship Operation Simulation.—An event-oriented simulation representing the operation of a commercial shipping fleet was developed at the request of the Maritime Administration. The event occurrence is

the entry of a ship into port for cargo to be discharged and loaded. The simulation can consider several networks to provide for the transshipment of cargo between adjoining networks. The simulation has proved useful in evaluating alternative shipping routines, schedules, and ship design configuration.

Progress in Transportation Simulation Studies.—Significant refinements in computer simulation models were made in the continuing studies of the Northeast Corridor Transportation Project of the Department of Transportation. Flexible preprocessors and post processors have been developed which aid in the application of the computer models to transportation planning analysis. Modification of new methods for evaluating interurban transportation studies has advanced to the point where run-teams have been organized to assemble all the models developed for the entire Northeast Corridor system, and to exercise the complex of models in solving transportation planning problems.

Safeguarding Fissionable Nuclear Materials.—The advent of private ownership of fissionable nuclear material beginning in 1970 creates a need for methods to prevent possible diversion of these materials to other than peaceful uses. One problem is to determine the level of material losses which is acceptable in nuclear fuel process systems, such knowledge being necessary in accounting for total disposition of the fissionable materials. NBS systems analysts are studying all nuclear fuel process systems to develop a process model which will trace the flow of nuclear material through all stages of each process, and which will include a capability for assigning acceptable loss rates at each stage of the process.

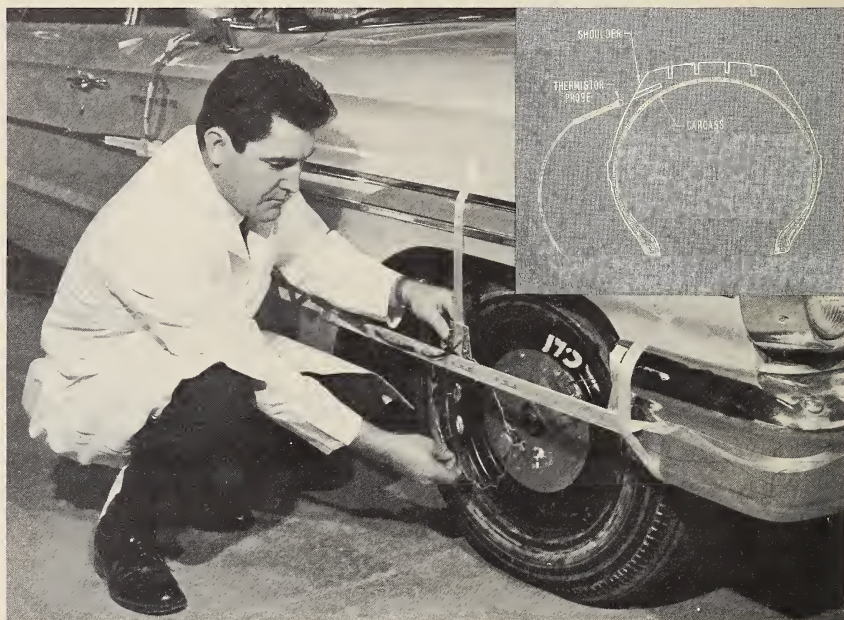
Demand Forecasting Models.—The IBS Operations Research Section collaborated with the IAT Technical Analysis Division in logical analysis of and numerical experimentation with variants of several travel-demand forecasting models prepared by contractors for the Department of Transportation. The result was improved model versions in a number of cases, as well as significant computational simplifications. Such work has great value in providing a better basis for planning the extensive transportation networks of the future.

Motor Vehicle Safety

During FY 1968 the Bureau continued to provide technical assistance to the National Highway Safety Bureau (NHSB) of the Department of Transportation in its development of motor vehicle safety performance standards. Research is being performed in the areas of tire systems, occupant restraint systems and braking systems.

Tire Systems

A significant breakthrough was made in the development of instrumentation to measure the temperature of a running tire on a vehicle. Thermistors or thermocouples embedded in the tire carcass are connected to a miniaturized solid-state transmitter mounted on the wheel rim. To assist the consumer in making an informed choice in the purchase of tires, methods are being developed to measure the speed capabilities, traction, mileage, impact resistance, and endurance properties of tires. Extensive road and laboratory testing of a large sample of passenger car tires will serve as a technical data base for this uniform quality grading system.



Temperatures created in tires during use are measured by a thermistor inserted in the tire shoulder. Antenna being mounted on car is part of a radio telemetry system used in the heat measurement.

Occupant Restraint Systems

The important research program involving human subjects continued at Holloman Air Force Base. The performance of 1968 model year production seats and restraint systems under collision conditions using anthropomorphic dummies was evaluated on the Daisy Decelerator at this facility. An important observation made during the research conducted on human subjects was the amount of restraint contributed by the legs during a 15 g deceleration accounting for as much as 55

percent of the subject's kinetic energy. An improved laboratory test was developed to measure more realistically the abrasion of seat belt webbing occurring during use.

Braking Systems

A laboratory for the evaluation of complete braking systems and their components is nearing completion. In operation are two types of brake lining testing machines, a Friction Materials Test Machine (Chase-Link type) and a Friction Assessment Screening Test Machine (FAST). A special purpose double-ended dynamometer is ready for installation and check out. It is capable of testing complete braking systems of motor vehicles up to light size trucks, including the new skid limiting devices. A popular American make sedan with front wheel disc brakes has been instrumented and is being used for correlation of vehicle road performance and laboratory testing. Laboratory test procedures for evaluating hydraulic brake fluids are being critically examined with respect to actual service performance requirements. Safety requirements were developed for standards relating to the packaging and labeling of brake fluids.

Engineering Materials

IAT provides advisory, consultative, and laboratory investigation services relating to selected engineering materials. The laboratory work is mainly for the development of test procedures which can be used by other testing laboratories. The staff responsible for this program works with national and international groups to establish standards for these materials. Services also include establishing a technical base for other Government agencies to set purchase specifications.

Magnetic Tapes for Satellite Recorders.—One of the most important causes of NASA satellite malfunctions is the early failure of flight tape recorders. NBS has undertaken studies of the changes of physical and chemical characteristics of tapes during use and also of the interaction of tapes and recording head surfaces. Advances have been made in two areas. Previously unused techniques have been applied to measure such properties as stiffness, surface hardness, and surface energy of tapes. The detection of very small amounts of foreign material on head surfaces by contact angle measurements, fluorescence, and infrared absorption spectroscopy were included in the study. Also, an informal liaison with satellite tape recorder manufacturers, magnetic tape manufacturers, and a number of NASA engineers concerned with satellite tape recorder problems has been established and NBS is gradually becoming a clearinghouse for information related to satellite magnetic tape and materials problems.

Evaluating the Stability of Record Papers.—The stability of paper is greatly dependent on the chemical modifications of the cellulose that occur during the manufacturing process, and on the interaction of cellulose with additives such as papermaker's alum (aluminum sulfate). Sensitive methods for the estimation of aluminum and of sulfate have been adapted to paper pulp-aluminum sulfate systems in order to ascertain the distribution of aluminum and of sulfate, and the effect of this distribution on stability. As file copies of most letters and manuscripts are made on manifold (carbon copy) paper, a special study was made of the stability, as measured by laboratory aging, of a representative group of these papers. Although acidity, as measured by a standard pH method, is an important factor in predicting stability, the study shows that a pH value alone can be very misleading, and that some laboratory aging procedure must be used in conjunction with acidity measurements.

Stability of Record Paper.—Differential Thermal Analysis (DTA), a technique for studying the thermal behavior of materials as they undergo physical and chemical changes during heating, has been found useful in ranking record papers in order of their relative stabilities. Most papers and related carbohydrate materials exhibit a typical decomposition pattern, and the temperature at which one of the peaks occurs appears to be related to stability, as measured by laboratory aging. This can be correlated with the acid content of the paper. A study of the effect of chemical modification of cellulose on the DTA pattern is in progress. In another series of tests, crosslinking of cellulose, as measured by the development of wet strength, has been found to occur during laboratory aging, and there is some evidence that crosslinking occurs during natural aging. This could have an important bearing on the evaluation of changes in the physical properties of paper during aging. Data could be interpreted as indicating degradation when, in fact, they might represent a change in the chemical structure that would not be related to stability.

Measuring the Durability of Paper.—The durability of paper is an elusive property that is difficult to measure in the laboratory. Some progress has been made, however, in the evaluation of durability through the development of a flexing device that uniformly destroys the physical structure of the paper. The rate of destruction, as measured by changes in rheological properties due to flexing, may be taken as a measure of relative durability. For specific end uses, durability can be greatly improved by incorporation of specially selected additives in the paper.

Precision of Standard Paper Test Methods.—As part of a study of the precision of standard methods used in the paper industry, an evaluation was made of the precision of the bursting strength method from data published over the past 40 years. It was found that no significant change in within-laboratory precision had occurred during this period. It was concluded that one or more standard reference materials would be needed to effect a substantial increase in precision.



Human subject slams into the water brake of the Daisy Decelerator in a simulated 15 g automotive collision. He is restrained by automotive lap belt and shoulder harness on the NBS experimental seat. Data recorded includes seat belt load, sled velocity and deceleration and human kinematic motion.

Flammable Fabrics Research.—The amended Flammable Fabrics Act broadened the coverage of items to include fabrics used in interior furnishings as well as those used in wearing apparel. Data acquired by NBS under the terms of the original Act, and information on individual cases gathered by the HEW Public Health Service are under review to determine whether new or revised standards and regulations are needed to protect the public. Tests on fabrics other than those used for clothing have been initiated as part of the program to see that the standards not only cover furnishings, but are also kept current in terms of items on the market.

Engineering Standards

Product Standards.—During 1967 the Institute broadened its efforts to provide improved technical services to the public and to State and Federal Government agencies in product standardization. It operated a Testing Laboratory for the General Services Administration's Federal Supply Agency which conducted work leading to more meaning-

ful specifications for commodities purchased by the Government. Product standards were revised by the Office of Engineering Standards Services to meet current needs of the consumer and industry.

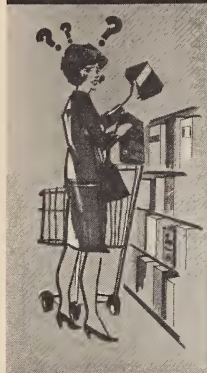
Amendments to the procedures for developing voluntary product standards were formally announced in May 1968. These provided for the more effective development of standards, particularly those relating to the quantities in which consumer products are sold at retail, and restated the numerical percentages definition of a consensus determination. During FY 1968, five proposed standards were circulated for comment, and 24 others were transmitted for committee approval of which 14 were later recommended for final acceptance. The review of older standards continued and 155 were examined to determine whether they should be withdrawn, revised, or revalidated. The collection of published standards has been expanded and now includes between 15,000 and 20,000 individual items which are being listed for publication in a reference index. These standards represent the output of more than 350 standards-making organizations or other groups.

Fair Packaging and Labeling.—During FY 1968 staff members were intensively engaged in carrying out NBS responsibility under the Fair Packaging and Labeling Act. With the cooperation and assistance of State and local weights and measures officials, other Federal agencies, and industry trade associations and committees, the Office of Weights and Measures investigated some 135 different types of packaged consumer commodities for evidence of undue proliferation. One hundred major industries are in various stages of a process to reduce and simplify the quantities in which consumer commodities are packaged. The packaged goods produced by these industries account for more than 60 percent of annual consumer expenditures for products covered by the Fair Packaging and Labeling Act and firm commitments have been received from about one third of them.

An important aspect of the fair packaging and labeling activity is to make sure that the customer can easily find and read the statement of net quantity on the package. Legibility depends among other things on the contrast between the color of the print and that of the background. A method of stating the color-contrast requirement, suitable for inclusion in packaging regulations, and capable of being used as a guide by graphic artists engaged in labeling design, has been prepared by IBS mathematicians for the NBS Office of Engineering Standards Services.

New State Standards.—This fiscal year NBS presented new State standards to nine States—Delaware, Connecticut, Ohio, Illinois, Tennessee, New Mexico, Utah, California, and Oregon. (The tenth State, Kentucky, was scheduled to receive standards officially on July 19, 1968.) Provision is being made for ten additional States to receive

END SLIDE-RULE SHOPPING



TYPICAL REDUCTIONS

SALAD AND
COOKING OILS



15



7

CEREALS



33



16

DETERGENTS



24



6

TOOTHPASTE



57



5

PAPER TOWELS



33



8

AIM:

CHOICE—Yes
CHAOS—No

INDUSTRY COOPERATES:

- 14 HAVE ACTED
- 65 PLAN CHANGES



Typical results of voluntary steps by industry to reduce the number of product package sizes since enactment of the Fair Packaging and Labeling Act on July 1, 1967. Industry has taken steps to cut the number of package sizes of 14 basic items. Eleven of these have made specific reductions of 289 sizes to 104. Sixty-five other industries have informed the U.S. Department of Commerce they are working on plans to consolidate package sizes.

standards—Maine, Pennsylvania, West Virginia, North Carolina, Georgia, Florida, Wisconsin, Missouri, Arkansas, and Hawaii—and manufacture of the third set of precise physical standards and instruments is underway. Recipients of the latest set will include: Vermont, New Jersey, Alabama, Indiana, North Dakota, Oklahoma, Texas, Wyoming, Idaho, and Maryland.

Basic laboratory training (two weeks) has been completed for 15 State laboratory technologists at various State laboratories and at the NBS Weights and Measures Laboratory. The first Regional Laboratory Training Seminar (intermediate training) at Madison, Wisconsin, attended by technologists from Ohio, Illinois, and Wisconsin, was also conducted by the Office of Weights and Measures.

Technical Training.—During the past fiscal year, 25 training courses were conducted throughout the country for State and local weights and measures officials. A one-week seminar was held at NBS for officials having supervisory responsibilities, and over 40 officials representing 33 jurisdictions attended. Other forms of training conducted by NBS officials included: (1) assistance to the States in field testing operations, (2) special purpose training for foreign visitors, Federal agencies, and private industry, and (3) training of State laboratory personnel (at both the State lab and NBS).

National Conference on Weights and Measures.—The 53rd National Conference on Weights and Measures held in Washington during June, accomplished a task of considerable magnitude in adopting a Model State Packaging and Labeling Regulation. The development of such a Model Regulation was complicated by the existence of individual regulations covering packaged consumer commodities issued by the Food and Drug Administration and the Federal Trade Commission, both pursuant to the Fair Packaging and Labeling Act. The States, with their obligation to provide labeling standards for all packages, found it necessary to create a document that included the stipulations of the two Federal regulations and, in addition, wholesale and institutional packages. Under the terms of the Fair Packaging and Labeling Act, requirements of the States with respect to consumer package quantity labeling cannot be less stringent than or inconsistent with the Federal requirements.

The Conference heard Lawrence C. McQuade, Assistant Secretary of Commerce for Domestic and International Business, report on vital economic developments within the Nation, and Dr. A. V. Astin, Director, National Bureau of Standards, discuss activities of the Bureau during the past year. Present were delegates from 40 States and the District of Columbia and, in addition, many businesses, industries, and consumer groups interested in weights and measures supervision.

TECHNOLOGICAL INNOVATION AND DIFFUSION

Technological innovation may be defined as that process whereby means are devised and applied for stimulating new technologies, channeling them in promising useful directions, and exploiting their use for purposes other than those for which they were originally developed. Technological diffusion, in the context of the IAT programs, is largely restricted to disseminating the results of Government sponsored R&D.

Invention and Innovation

The Office of Invention and Innovation is the IAT activity which helps to develop an environment conducive to technological change. Its basic program has three aspects: providing a national basis for formulation of climate-setting Federal policies, offering assistance to inventors, and education.

In FY 1968, the Office assumed responsibility (as assigned by the Assistant Secretary of Commerce for Science and Technology) for the program, planning and budgeting activity of the Department of Commerce Technology Program. Department programs included in this responsibility are the Patent Office, Office of State Technical Services, the NBS Clearinghouse for Federal Scientific and Technical Information and the Office itself.

Invention Policy.—The Office of Invention and Innovation continued to assist other organizations (such as the Organization of American States, the Arms Control and Disarmament Agency, Committees of the U.S. Senate) to further the work of the Commerce Secretary's Panel on Invention and Innovation. Seminars for universities and professional organizations have been held throughout the country. A new Secretarial panel on the problems associated with the allocation of venture capital has been established. The National Inventors Council has embarked on a comprehensive assessment of the inventor and his relationship to society.

Invention Programs.—The number of states holding invention expositions and inventors' seminars increased to 24 during fiscal 1968 and several other states are planning to join this effort during the coming year. The programs are organized and conducted by state organizations and universities and are aimed at facilitating the licensing, development, and exploitation of inventions. NBS provides assistance in planning and undertaking the expositions and also provides speakers and experts for the seminar programs.

Education for Innovation.—The Bureau is working with the American Society for Engineering Education and the National Science Foundation to survey opportunities for encouraging innovative design projects in the engineering schools. The program started from recommendations of the National Inventors Council and at this stage is directed primarily to an analysis of opportunities for bringing public sector problems into engineering curricula.

The Clearinghouse

A second major activity in the category of Technological Innovation and Diffusion is the IAT program for the dissemination of technical information. This program centers around the Clearinghouse for Federal Scientific and Technical Information which is the central point of contact in the Federal Government for disseminating the results of Government-sponsored R&D to industry, commerce, and the general public. In addition to the Clearinghouse, the Institute maintains certain specialized technical information centers covering engineering standards, weights and measures, instrumentation, and computer sciences.

The output of the Clearinghouse increased substantially during FY 1968. The Clearinghouse journal *U.S. Government Research and Development Reports (USGRDR)* announced 37,100 titles. There are now over 547,000 titles in the Clearinghouse collection. Also, over 2 million copies of research reports were distributed to more than 70,000 customers.

Document Announcement.—The Clearinghouse began announcing unclassified/unlimited Department of Defense technical reports to Defense Documentation Center (DDC) users through the *USGRDR*. Under a DDC/Clearinghouse agreement all registered DDC users receive one copy each of *USGRDR* and its companion publication *USGRDR INDEX*.

In January 1968 the title of the *USGRDR/Index* was changed from *Government Wide Index to Federal Research & Development Reports (GWI)* to *U.S. Government Research & Development Reports Index (USGRDR-I)*. Changes in the format of the journal and index were made to improve presentation and readability.

The publication of the separate announcement journal *Technical Translations* terminated with the December 30, 1967 issue. Technical translations are now announced in the *USGRDR*.

In an experiment with a new announcement mechanism, *Clearinghouse Announcements in Science and Technology (CAST)*, over 1,000 Air Force technical personnel and NBS scientists participated in a test as subscribers to a method for scanning the flow of information in 46 separate fields of technology. Reports resulting from research in defense, space, nuclear energy, transportation, area development, education, and other national programs were included. More than 85 percent of the participants reported that CAST opened up new information sources and saved them valuable time. The results of the experiment, which ran for 6 months, will be used in the development of a new Clearinghouse announcement medium beginning in FY 1969.

New Document Input.—The Clearinghouse expanded its coverage in acquiring technical information from Federal agencies and from private organizations. Documents were acquired from the Department of Transportation and the British Road Research Laboratories on highway safety research; from the Department of Housing and Urban Development on urban transportation; from the Office of Economic Opportunity on research in the social sciences and summaries of Federal programs for FY 1967 in each State; from the Federal Communications Commission on magnetic tapes containing radio station licensee data; from the Federal Aviation Agency on aircraft accident reports and briefs of accidents in the field of U.S. Civil Aviation. In the private sector reports were received on area development planning from the American Institute of Planners and the Metropolitan Council of Governments.

Clearinghouse Computer.—An IBM 360-20 computer was installed which is programmed to handle processing of orders received for documents.

CENTER FOR RADIATION RESEARCH

The Center for Radiation Research (CRR) was created in March 1968 by consolidation of the Radiation Physics Division of the Institute for Basic Standards with the Reactor Radiations Division of the Institute for Materials Research. With this action, the extensive radiation facilities at NBS were placed under a single management with responsibilities different in some respects from those of the three Bureau Institutes. Because the Center contains unique facilities and competences, it not only has the responsibility for carrying out the Bureau's mission in the area of radiation, but also for making available its resources and capabilities to other Government agencies, industry, and universities. This policy of facility sharing ensures efficient utilization of large multi-purpose facilities such as the reactor and linear accelerator by making them available to a broad segment of the scientific community.

Radiation of concern to CRR is that produced by radionuclides, reactors, and particle accelerators with energies between 5 keV and 300 MeV. In this range, the Center provides national leadership in accurate and uniform techniques of measurement of radiation and its interaction with matter, and serves as a national resource providing facilities and competence for the development and use of radiation for investigating properties of atoms, nuclei, and materials. Current programs can be grouped roughly as follows:

1. Radiation Measurement and Standards
2. Nuclear Physics Research
3. Facilities Operations
4. Technical Assistance to Others

Approximately 20 percent of the Center's work is supported by other Government agencies.

Radiation Measurement and Standards

Neutron Source Standards.—The precision of the calibration service for neutron sources by the manganous-sulfate bath method has been improved from a random uncertainty of 0.6 percent to 0.2 percent. This was accomplished by improving the manganese-56 detection system. The new system continuously counts the saturated bath by circulating the activated solution past a gamma scintillation detector which is shielded from the neutron source.

Neutron Energy Transfer.—Calculations of energy transfer to matter by neutrons from thermal energies to 18 MeV have been carried out for a number of important compounds and mixtures including tissue. These calculations differ from nearly all previous calculations in that all detailed neutron reaction processes are included. The work has demonstrated the importance of usually neglected neutron-induced charged-particle reaction processes, which are very effective for delivering neutron energy to materials.

Neutron Cross Sections.—The total neutron cross sections of iron, carbon, and lead have been measured in the neutron energy range 0.7 to 4.4 MeV. The neutron time-of-flight method was employed, with the NBS electron linear accelerator used as the pulsed neutron source. The energy resolution varied from 0.4 percent to 2 percent; the overall uncertainty on the absolute cross section values was ± 2.5 percent, and the statistical accuracy of each point was about ± 1.5 percent. The results are in general agreement with earlier data from other laboratories. The measurements revealed much new fine structure in iron and lead, as well as a new resonance in carbon. Data are now being used for shielding calculations at other Government laboratories, where they have helped to resolve some annoying discrepancies.

Response of Silicon Detectors to Monoenergetic Electrons.—The response of silicon semiconductor detectors to monoenergetic electrons in the energy region from 0.25 to 1.00 MeV has been systematically investigated. This was accomplished by exposing silicon transmission detectors to normally incident electrons with energies of 0.25, 0.50, 0.75, and 1.00 MeV. When the detector thickness was less than the incident electron range, the pulse-height distributions produced by electrons were characterized by an absorption peak and a relatively broad escape peak which is associated with transmission and reflection of electrons. Taking into account the resolution of the detectors, which were used at room temperature, comparisons were made between the experimental pulse-height distributions and Monte Carlo results calculated for the same conditions. Good agreement was found between theory and experiment at 0.75 and 1.00 MeV; however, unexplained differences appeared in the position of the escape peak at 0.25 and 0.50 MeV.

High-Energy Electron and Photon Absorbed Dose Standard.—A project was initiated to establish a service for calibrating measuring instruments in terms of absorbed dose produced with monoenergetic electron and bremsstrahlung beams between 1 and 50 MeV. A new calorimetric design and measuring technique was devised to reduce or eliminate uncertainties caused by temperature gradients, a basic problem affecting accurate calorimetric measurements. Although the

calorimeter was intended for measuring absorbed dose, the principles are applicable to calorimetry in general. Calculations concerning the performance of the calorimeter revealed a mathematical identity concerning heat transfer and its electrical analog.

Cobalt-60 Source Calibration.—The total power output of a cobalt-60 gamma ray source furnished by the National Research Council, Ottawa, Canada was determined. A total absorption calorimeter was used for the measurement. An accuracy of 0.1 percent was obtained. The source will also be measured at the national laboratories in Canada and the USSR and at the International Bureau of Weights and Measures in order to assess the measuring capability of the four laboratories.

Solid State Dosimetry.—An investigation was carried out of the temperature dependence of photocurrents produced by x and gamma rays in silicon radiation detectors of different types used for exposure rate measurements under the dc mode of operation. The different temperature dependence of short-circuit currents produced in p-n, p-i-n, and surface barrier type detectors was found to be due to the different structure of these detectors rather than to a different temperature dependence of their basic electronic properties. The results of this investigation indicate the possibility of developing an exposure rate meter with silicon radiation detectors showing only a very small temperature dependence over a chosen temperature range.

Ferrous-Sulfate Dosimeter Studies.—Since July 1967 the Bureau has provided a quarterly service of mailing Fricke dosimeter units to those requesting assistance with absorbed-dose measurements in high-energy electron beams. The purpose is to promote uniformity of absorbed-dose calibrations among the users of electron beams in the energy range 5 to 50 MeV. In connection with this service, experience has been gained in the preparation, shipping, and evaluation of large numbers of Fricke dosimeters, and information has been gathered on the types of high-energy electron machines in use in this country. Results of the studies indicate there is a reasonable degree of uniformity among participating users of high-energy electron beams, with only a few exceptions.

Radioactivity Standards.—During the past year, 366 radioactivity standards were sold, 36 source calibrations were performed, and the complete stock of radium solution standards was recalibrated. New standards produced were a cadmium 109–silver 109 “ γ -ray kit” point source, a tin 113–indium 113 γ -ray solution source, and a 10^{-8} g radium source in solution for radon testing. Radium standards in solution form have been reissued at the 10^{-9} g radium level. The half-lives of

two states in neptunium-239 have been measured and a new method of calibration for the disintegration rate of cadmium 109 has been developed utilizing a thin-walled well-type scintillation crystal. Procedures have been developed which enable the determination of activities by means of scintillation spectrometers to accuracies of the order of 1 percent. The ^{113}Sn radioactivity standards (with an uncertainty of ± 2.2 percent) were produced with the aid of these techniques. A determination of radioactivities present in certain Antarctic soil by means of gamma-ray spectrometry was made.

The Bureau participated in experimental work in connection with the development of an ASTM standard procedure for determination of radium in water, as well as the intercomparison of cobalt 57 sources among pharmaceutical laboratories and national laboratories in the U.S. and Canada.

Electron Interactions.—The revamped 500-keV electron accelerator has been used for measurements on the response of thermoluminescent materials to electrons, and on atomic binding effects in Möller scattering. The latter experiment revealed prominent, but theoretically unpredicted, structure in the inelastic spectra and increasing agreement with the Möller cross section for increasing atomic numbers.

Other measurements were made on electron response to radiation-sensitive dye systems and work is currently being pursued on x-ray spectral data, especially those related to cross section or production efficiency determinations of characteristic radiations.

Monitoring of High Energy Electron Beams.—A nonintercepting technique for measuring the integrated current of pulsed high-energy electrons is being developed, employing ferrite toroids as pulse transformers. The system provides stable and reproducible results when compared with a Faraday cup. Nonlinear effects due to changes in pulse length and amplitude are being investigated. A simple, radiation hard beam position monitor has been developed. The sensitivity is sufficient to permit beam position measurements with an accuracy of better than 1 mm at pulsed beam currents less than 1 mA.

Nuclear Physics Research

Electron Scattering.—Experiments in progress include inelastic scattering from ^{12}C , concentrating on the excitation region 17–27 MeV; inelastic scattering from low-lying levels of ^{16}O ; and an experiment attempting to measure the charge form factor of the proton to very high accuracy at low momentum transfer. Collaborative research programs using the NBS electron scattering facility are in progress with research teams from five universities. The multidetector “ladder” counter system employing semiconductor detectors has been brought into operation with excellent results. A number of other laboratories

throughout the world are adopting similar systems for their electron scattering programs. The design aim of an overall resolution capability of 0.1 percent has been achieved. At this resolution, experiments can be performed with electron beam current of about $3\ \mu\text{A}$.

Heavy-Ion Spectrometer Experiments.—The electron and photon disintegration cross sections of ${}^4\text{He}$ are being measured with the NBS heavy-ion spectrometer and NBS linac. The mass identification feature of the heavy-ion spectrometer makes possible an unambiguous determination of the ${}^4\text{He}(\gamma, p)T$, ${}^4\text{He}(e, pe')$ and ${}^4\text{He}(\gamma, n){}^3\text{He}$, ${}^4\text{He}(e, ne'){}^3\text{He}$ cross sections. This measurement will substantially improve the precision of total and different (in angle) cross sections from threshold to 50 MeV for proton reaction and from 24 MeV to 100 MeV for neutron reaction.

The Nuclear Three-Body Problem.—Calculations of the photodisintegration of ${}^3\text{H}$ and ${}^3\text{He}$ have been extended using a more consistent model based on a ground state wave function that is an exact solution of the three-body Schrödinger equation with separable potentials of the Yamaguchi type. The final states contained interactions between nucleon pairs. Calculations were carried out for both the Yamaguchi-Sitenko-Kharchenko and Tabakin sets of interaction parameters. Since the wave functions used have also been employed to describe the scattering of neutrons by deuterons, the theoretical model serves as a means of relating two types of experimental data. Investigations are underway to check the validity of the model for calculating the three alpha particle break-up of ${}^{12}\text{C}$.

In a separate investigation the problem of constructing a practical calculational scheme for off-energy-shell two-body amplitudes was undertaken which avoided the use of integral equations. The off-energy-shell two-body amplitudes are used as input to the Faddeev formulation of the three-body problem. It was found that the calculation could be done with a differential equation similar to the Schrödinger equation but containing an extra inhomogeneous term.

Nuclear Structure Studies by Electron and Photon Excitation.—The program to investigate nuclear states by exciting them with electrons and photons is continuing. Nuclear levels in the isotope ${}^{179}\text{Hf}$ have been investigated and a report on this nucleus is being prepared. Further levels in isotopes of Au and Ir are being investigated and other elements will be studied in the future. An apparatus for investigating elastic and inelastic scattering of electrons at 180° has been installed and should be operating shortly. This apparatus will provide experimental tests of established nuclear force and structure theories by investigating how low atomic number elements behave in interactions where non-nuclear excitation scattering is at a minimum.

Large Angle Inelastic Electron Scattering.—Data taken on the inelastic scattering of 200- and 400-keV electrons at large angles from various targets has been analyzed and a report published. These results show disturbing deviations from the available theoretical calculations and are being investigated further. In particular, the incident electron energy is to be extended from 400 keV upwards to 4 MeV by utilization of the 1.5 and 4.0 MeV NBS electron accelerators.

Nuclear Spectroscopy.—The asymmetry of beta particles from oriented thulium 170 and praseodymium 142 nuclei implanted in iron foils by the NBS isotope separator has been used to measure the effective internal magnetic fields for these nuclei. Beta spectroscopy under the conditions of cryogenic nuclear orientation has been aided by the development of thick silicon detectors and preamplifiers which function well at temperatures of 1.5 K. The effect of polarizing fields, foil angles, and scattering on electron spectra have been investigated. Optimum conditions for the apparatus have been determined for sustained maximum orientation and for accurate thermometry using the anisotropy of a ^{57}Co gamma ray measured by germanium detectors.

The Photonuclear Data Center.—The first Supplement to Miscellaneous Publication 277, Photonuclear Data Index, was published and circulated to workers in the field. This supplement covered data added to the Center's files in the period January 1, 1965 through April 1967. Each entry in this index supplies quantitative data about the specific type of experimental information given in each reference to the literature cited. The complete index to data in the Center's files has now been put on magnetic tape, making possible automatic searches using a small computer, for reference to any one or all of nine specific types of information.

Facilities Operations

Linear Accelerator.—During fiscal year 1968 in excess of 1500 hr of beam time were provided for experiments using the NBS linac. This was 57 percent of the scheduled beam time. Numerous modifications to the accelerator and beam-transport equipment have been accomplished to improve beam quality and reduce maintenance problems. The chief maintenance problems continue to be associated with the fact that round-the-clock operation is not possible. Despite this, beam has been provided at under \$200 per hour of beam-on-target time.

Experiments using the accelerator include programs in electron scattering, photoneutron production, production of heavy ions, measurement of neutron total absorption cross sections, studies in beam monitoring, programs in activation analysis, and production of radioactive sources for materials studies using Mössbauer sources and for

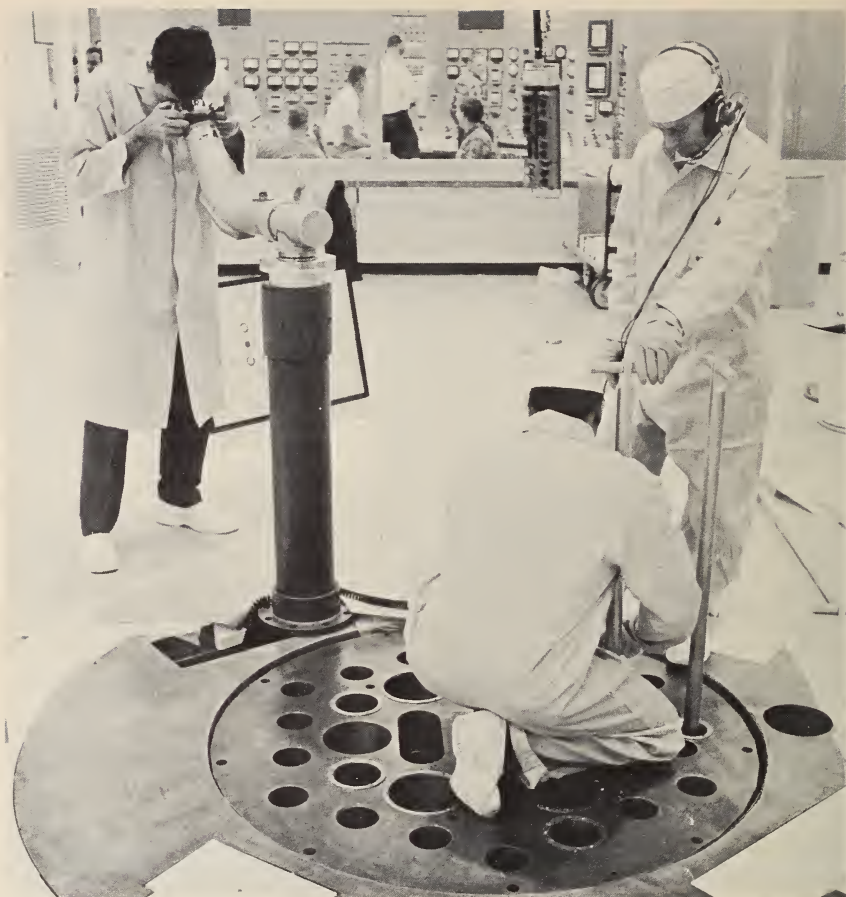
studies of photonuclear reactions. Programs in electron and x-ray beam dosimetry, accelerator research, and production and use of mono-energetic positron and photon beams are in preparation. Collaborative programs with groups from six different universities are or will soon be in operation.

A major improvement during the year for time-of-flight experiments has been the development of enhanced short pulse linac operation. Beam pulse lengths of three to five nanoseconds and beam currents in excess of two amperes at the end of the accelerator are now possible. Delivery of energy analyzed beams (2 percent) in excess of one ampere to experiments with these pulse lengths is routine.

NBS Reactor.—On December 7, 1967 the National Bureau of Standards Reactor (NBSR) achieved criticality for the first time. This was the culmination of more than nine years of planning and over four years of construction. The NBSR, a high-flux, tank type, heavy water cooled and moderated machine will operate initially at a power of 10 MW (thermal) and will have a flux of 10^{14} n/cm²/s. Several months of low power testing are required to determine the characteristics of the reactor before operating at high power. When NBSR becomes fully operational, it will be used for neutron diffraction and scattering experiments in the area of materials research and for studies in radiochemistry.

4 MeV Accelerator.—The experimental flexibility of the NBS 4-MeV Van de Graaff accelerator has been increased by the addition of a new 18-in. diameter electron scattering chamber. This chamber, which may be used for electron scattering experiments, dosimeter irradiations and charged particle detector calibrations, provides such features as a remotely operated target viewing system to determine incident beam position, scattering ports every 30° from 0 to 180°, and complete electrical insulation for current measurements. The accelerator system has also been equipped with a beam centering feedback system to automatically maintain beam location, independent of small changes in the operating parameters of the accelerator. Further updating of the accelerator voltage stabilizing system is also underway.

Isotope Separator.—The new electromagnetic separator has been used for the preparation of pure, thin, mass-identified sources of stable and radioactive isotopes. Sources which were made with the isotope separator were used in Mössbauer, photonuclear reaction, nuclear orientation, and range distribution studies. The ion implantation of sulphur in semiconductors such as gallium arsenide and cadmium sulfide resulted in the production of materials with desirable properties. An investigation of the radiation damage to metal chelates has been started.



Reactor operators using fuel handling tools to lock fuel elements into the core of the NBS reactor. Control room can be seen in the background. Technician on left is observing the operation in the core through a periscope.

Synchrotron Move.—During this year the major effort of moving the NBS 180-MeV synchrotron from the Washington site to Gaithersburg was essentially completed. Efforts are now underway to achieve or better the beam currents previously available before experimental programs using the accelerator resume.

Technical Assistance to Others

Studies for Other Agencies

Standard Nuclear Instrument Module System.—The standard Nuclear Instrument Module (NIM) System, developed in conjunction with the National Laboratories of the U.S. Atomic Energy Commission, has had a tremendous impact on the entire nuclear instrumentation field and has revolutionized the manner in which experiments are performed in nuclear physics. Practically all modular nuclear in-

strumentation currently produced in the United States is in accordance with this standard. The NIM System is also the dominant modular nuclear instrumentation system worldwide. The NIM System development was undertaken as a result of an NBS report issued in December 1963 that pointed out interchangeability problems that accompanied the advent of transistorized modular instruments and recommended that a coordinated effort be initiated to produce a standard module system to reduce these difficulties. Specifications for standard modules to assure mechanical and electrical interchangeability were drawn up in 1964; laboratory and industry utilization began at the end of that year. By 1966 the NIM System had been adopted by practically all U.S. nuclear instrument manufacturers and was responsible for a considerable export market in nuclear instruments.

Radioactive Contamination of Materials.—An extensive study is being carried out on behalf of the USAEC to determine the extent of low-level contamination in chemical reagents and commercial materials, including the study of variations with time, nature, manufacturing practice and geographical origin. Over 500 samples have been studied to date.

Conferences

Molecular Dynamics and Structure of Solids Conference.—The Molecular Dynamics and Structure of Solids Conference was held at the National Bureau of Standards site in Gaithersburg, Md., on October 16–19, 1967. Sessions were attended by approximately 250 people from Government, industry, and universities. Objectives of the symposium were to:

Encourage interdisciplinary cooperation by demonstrating the correlation of various techniques applied to the study of molecular dynamics and structure of solids.

Stimulate among participants an appreciation of techniques outside their own specialties.

Promote a more complete understanding of the materials of interest.

Neutron Cross Sections and Technology Conference.—The second conference on “Neutron Cross Sections and Technology” was held at the Shoreham Hotel, Washington, D.C., on March 4–7, 1968. Approximately 350 persons from Government, universities, and industry attended the Conference. The purpose of the second conference was to provide a common meeting area for the exchange of information among nuclear scientists and engineers interested in neutron cross sections. The conference program was designed to consider all aspects of neutron cross sections, the need for accurate measurements, and their determination by theoretical and experimental techniques and their applications.

APPENDIXES

ORGANIZATION OF THE NATIONAL BUREAU OF STANDARDS ¹

The Bureau is headed by a Director who is appointed by the President with the advice and consent of the Senate. The Director is assisted in the overall management of the Bureau by a Deputy Director. In addition there is an Associate Director for Administration who is responsible for the planning and operation of facilities and of administrative management services in support of the Bureau's technical programs.

Technical program activities are conducted in three Institutes and a Center for Radiation Research. Each is headed by a Director who is responsible for the development and direction of research programs and central national services essential to the fulfillment of a broad segment of the Bureau's mission. These major organizational units are:

(1) The Institute for Basic Standards, which includes 12 divisions (5 in Boulder, Colo.), each serving a classical subject matter area of science and engineering; also 3 administrative divisions serving technical divisions located in Boulder, Colorado;

(2) The Institute for Materials Research, which consists of 6 divisions, organized primarily by technical field;

(3) The Institute for Applied Technology, which includes 16 divisions oriented to high-technology industries; and

(4) The Center for Radiation Research, which includes 4 divisions concerned with the theory and application of radiation.

DIRECTOR
ALLEN V. ASTIN

DEPUTY DIRECTOR
I. C. SCHOONOVER

Assistant to the Deputy Director
P. H. KRATZ

¹ As of June 30, 1968.

OFFICE OF THE DIRECTOR

Assistants to the Director

G. E. AUMAN

C. N. COATES

A. G. McNISH

Legal Advisor

A. J. FARRAR

Office of Industrial Services

G. S. GORDON

Office of Public Information

A. V. GENTILINI

Office of Academic Liaison

S. SILVERMAN

Office of Engineering Standards Liaison

A. A. BATES

Office of Program Development and Evaluation

R. D. HUNTOON

M. B. WALLENSTEIN

R. E. FERGUSON

Senior Research Fellows

C. EISENHART

K. SHULER

Technical Support Units Reporting to the Deputy Director

Coordinator of Special International Programs-----L. L. MARTON

Office of Technical Information and Publications-----W. R. TILLEY

Library Division-----E. L. TATE

Instrument Shops Division-----F. P. BROWN

Measurement Engineering Division-----G. F. MONTGOMERY

OFFICE OF ASSOCIATE DIRECTOR FOR ADMINISTRATION

R. S. WALLEIGH

Deputy Associate Director

P. H. SCHRADER

Patent Advisor

D. ROBBINS

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Administrative Services Division	G. W. KNOX
Budget Division	J. E. SKILLINGTON
Management and Organization Division	J. T. HALL
Personnel Division	G. R. PORTER
Plant Division	H. GRAHAM
Supply Division	G. B. KEFOVER

INSTITUTE FOR BASIC STANDARDS

Director
E. AMBLER

Deputy Director
Vacant

Deputy Director, Institute for Basic Standards/Boulder ¹
B. W. BIRMINGHAM, Acting

Coordinator for Measurement Services
H. L. MASON

Office of Standard Reference Data	E. L. Brady
Atomic and Molecular Data	Information Services Design
Chemical Kinetics	Mechanical Properties
Colloid and Surface Chemistry	Nuclear Data
Information Services	Solid State Properties
Thermodynamics and Transport Data	

Applied Mathematics	E. W. Cannon
Numerical Analysis	Statistical Engineering
Operations Research	Systems Dynamics

Electricity Division	C. H. Page
Resistance and Reactance	High Voltage
Electrochemistry	Absolute Electrical
Electrical Instruments	Measurements

Metrology Division	T. R. Young, Acting
Optics Metrology Branch	Length Metrology Branch
Photometry	Length
Image Optics and Photography	Engineering Metrology
Colorimetry and Spectrophotometry	Mass and Volume Branch
	Mass and Volume

Mechanics Division	E. C. Lloyd, Acting
Sound	Engineering Mechanics
	Rheology

¹ Located at Boulder, Colo.

Mechanical Measurements Branch	Fluid Mechanics Branch
Pressure Measurements	Fluid Meters
Vacuum Measurements	Hydraulics
Vibration Measurements	Aerodynamics
Humidity Measurements	
Heat Division -----	R. P. Hudson
Heat Measurements	Statistical Physics
Cryogenic Physics	Molecular Energy Levels
Equation of State	Temperature
	Radiation Thermometry
Atomic Physics Division -----	K. G. Kessler
Spectroscopy	Electron Physics
Infrared and Microwave	Atomic Physics
Spectroscopy	Plasma Spectroscopy
Far Ultraviolet Physics	
Radio Standards Physics Division ¹ -----	H. S. Boyne, Acting
Solid State Electronics	Quantum Electronics
	Plasma Physics
Radio Standards Engineering Division ¹ -----	H. M. Altschuler
H F Calibration Service	H F Electrical Standards
H F Impedance Standards	M W Calibration Service
M W Circuit Standards	Electromagnetic Field Standards
Time and Frequency Division ¹ -----	J. A. Barnes
Frequency-Time Dissemination	Frequency-Time Broadcast Services
Research	
	Atomic Frequency-Time
	Standards
Laboratory Astrophysics Division ¹ -----	L. M. Branscomb
Cryogenics Division ¹ -----	D. B. Chelton, Acting
Cryogenic Technical Services	Cryogenic Data Center
Cryogenic Properties of Solids	Properties of Cryogenic Fluids
Cryogenic Systems	Cryogenics Metrology
	Cryogenic Fluid Transport Process
Administrative Services Division ¹ -----	B. F. Betts
Supply	Office Services
	Drafting Services
Instrument Shops Division ¹ -----	R. S. Perrill
Instrument Shop I	Instrument Shop II
Welding-Sheet Metal Shop	Glassblowing Shop
Plant Division ¹ -----	E. A. Yuzwiak
Construction-Maintenance	Special Services
	Custodial Services Section

¹ Located at Boulder, Colo.

INSTITUTE FOR MATERIALS RESEARCH

Director
J. D. HOFFMAN

Deputy Director
H. E. SORROWS

Office of Standard Reference Materials-----W. W. Meinke

Analytical Chemistry Division-----W. W. Meinke

Radiochemical Analysis	Microchemical Analysis
Spectrochemical Analysis	Analytical Mass Spectrometry
Electrochemical Analysis	Organic Chemistry
Analytical Coordination Chemistry	Activation Analysis
Separation and Purification	

Polymers Division-----R. R. Stromberg, Acting

Polymer Dielectrics	Dental Research
Polymer Chemistry	Polymer Characterization
Polymer Crystal Physics	Polymer Interfaces
Molecular Properties	Thermophysical Properties

Metallurgy Division-----E. Passaglia

Engineering Metallurgy	Corrosion
Alloy Physics	Metal Physics
Lattice Defects and Microstructures	Electrolysis and Metal Deposition
Crystallization of Metals	

Inorganic Materials Division-----J. B. Wachtman

Inorganic Chemistry	Physical Properties
Inorganic Glass	Crystallography
High Temperature Chemistry	Solid State Physics Section

Physical Chemistry Division-----J. R. McNesby, Acting

Thermochemistry	Mass Spectrometry
Surface Chemistry	Photo Chemistry
Elementary Processes	Radiation Chemistry

INSTITUTE FOR APPLIED TECHNOLOGY

Director
L. M. KUSHNER

Deputy Director
Vacant

NBS/GSA Test and Development Division-----P. J. Franklin

Manager, Engineering Standards-----M. W. Jensen

Office of Engineering Standards Services-----D. R. Mackay

Product Standards Section	Information Section
---------------------------	---------------------

Mandatory Standards Section

Office of Weights and Measures-----	M. W. Jensen
Office of Invention and Innovation-----	D. V. DeSimone
Innovation Studies Program	Invention Programs
Engineering Education Program	
Office of Vehicle Systems Research-----	P. J. Brown
Clearinghouse for Federal Scientific and Technical Information----	H. E. Sauter
Document Distribution and Reproduction Branch	
Automated Systems and Services Branch	
Administrative Operations Branch	
Joint Publications Research Service	
Documents Processing Branch	
Product Evaluation Division-----	S. B. Newman
Plastics and Textiles	Paper Evaluation
Fibrous Systems	Fabric Flammability
Viscoelastic Materials	
Building Research Division-----	J. R. Wright
Structures	Materials Durability and Analysis
Fire Research	Codes and Standards
Scientific and Professional Liaison	Building Systems
Sensory Environment Branch	
Environmental Engineering	Psycho-physics
Building Transport Systems	
Electronic Instrumentation Division-----	M. G. Domsitz
Semiconductor Characterization	Instrumentation Applications
Electron Devices	Semiconductor Processing
Technical Analysis Division-----	W. E. Cushen
Corridor and Highway Studies	Economic Analysis
Systems Analysis and Human Factors	Development of New Methodology
Center for Computer Sciences and Technology-----	H. R. J. Grosch
Office of Information Processing Standards-----	J. O. Harrison, Jr.
Technical Information Exchange-----	M. R. Fox
Computer Services Division-----	W. B. Ramsay, Acting
Systems Research and Development Division-----	E. C. Marden
Information Processing Technology Division-----	J. P. Nigro

CENTER FOR RADIATION RESEARCH*

Director

C. O. MUEHLHAUSE, Acting

Deputy Director

R. S. CASWELL, Acting

Radiation Theory

Health Physics

Reactor Radiation Division-----	R. S. Carter, Acting
Reactor Operations	Neutron Solid State Physics
Engineering Services	Radiation Effects

*Reports to the Deputy Director, NBS

Linac Radiation Division-----	J. E. Leiss, Acting
Linac Operations	Photonuclear Physics
Radiation Physics Instrumentation	Electronuclear Physics
Nuclear Radiation Division-----	H. H. Landon, Acting
Nuclear Physics	Radioactivity
	Nuclear Spectroscopy Section
Applied Radiation Division-----	J. W. Motz, Acting
X-Ray Physics	Dosimetry

FIELD ESTABLISHMENTS

Institute for Basic Standards

Metrology Division Field Station :	
Visual Landing Aids Field Laboratory	Arcata, California
Time and Frequency Division Field Stations :	
Standard Frequency Station WWV-WWVL-WWB	Fort Collins, Colorado
Standard Frequency Station WWVH	Maui, Hawaii
Laboratory Astrophysics Division Field Station :	
Poor Man's Relief Mine, Four-Mile Canyon	Boulder, Colorado

Institute for Applied Technology

Office of Weights and Measures Field Stations :	
Master Railway Track Scale Depot	Clearing, Illinois

SUMMARY OF NBS STAFF AS OF JUNE 30, 1968

	Washington	Boulder	Total
Full-time permanent staff ¹ -----	2, 939	580	3, 519
Other staff ² -----	305	48	353
Total paid staff-----	3, 244	628	3, 872
Research associates—guest workers-----	131	16	147
Total NBS staff-----	3, 375	644	4, 019
Professional Staff With Academic Degrees:			
Physicists-----	395	114	509
Chemists-----	272	7	279
Engineers-----	172	89	261
Mathematicians-----	49	7	56
Other-----	128	5	133
Total-----	1, 016	222	1, 238

¹ Includes Post Doctoral Research Fellows.

² Summer, Youth Opportunity Corps, Part-Time, Intermittent and Temporary.

FINANCIAL DATA FOR FISCAL YEAR 1968

<i>Program and source of financing</i>	<i>Obligations incurred in thousands of dollars (rounded)</i>
Supported by NBS Appropriations	
Operating programs:	
Research and technical services.....	\$32, 283
Civilian industrial technology.....	139
Special foreign currency program.....	444
Construction and facilities programs:	
Plant and facilities.....	894
Construction of facilities.....	2, 996
Total obligations, NBS appropriations.....	36, 756
Supported by other funds ¹	
From other Federal agencies.....	24, 036
From other sources.....	4, 392
Total obligations, other funds.....	28, 428
Total program.....	65, 184

¹ Work supported by other funds consists of research and development programs for other Government agencies; consultative, advisory, and technical services, the performance of various tests and calibrations, and the manufacture and sale of standard reference materials for other Government agencies and the public.

RESEARCH ASSOCIATES AND GUEST WORKERS

Research Associates and Their Sponsors During Fiscal Year 1968

AiResearch Manufacturing Company

Simpson, Mr. Anthony U.

American Cyanamid Company

Hodgson, Dr. William G.

American Dental Association

Argentar, Mr. Harold

Bowen, Mrs. Joy C.

Bowen, Dr. Rafael L.

Brown, Dr. Walter E.

Carlson, Mr. Elmer T.

Caul, Mr. Harold J.

Gregory, Mr. Thomas M., Jr.

Kingsbury, Mrs. Pamela

Mabie, Mr. Curtis P., Jr.

Manuszewski, Mr. Richard C.

McDowell, Mr. Hershel

American Dental Association—Continued

Moreno, Dr. Edgard C.
Paffenbarger, Dr. George C.
Palcic, Miss Julia M.
Patel, Mr. Prafull
Wallace, Mrs. Betty M.
Waterstrat, Mr. Richard M.

American Electroplaters Society

Johnson, Mr. Christian E.

American Society for Testing and Materials

de Groot, Mr. Johan H.
Evans, Mrs. Eloise H.
McMurdie, Mr. Howard F.
Morris, Mrs. Marlene C.

American Society for Testing and Materials (Concrete and Concrete Reference Laboratory)

Anderson, Mr. Harry G., Jr.
Atkinson, Mr. George O., Jr.
Dise, Mr. John R.
Johnson, Mr. Marlin C.
Katz, Mrs. Anne K.
Liskey, Mr. John F.
Markus, Mr. Walter
McCarthy, Mr. Dennis D.
Seiler, Mr. James F., Jr.
Spring, Mr. Curtis B.
Sturm, Mr. William F.
Wallace, Mr. Dennis R.

American Viscose Division (FMC Corporation)

Oneal, Mr. Glen, Jr.

Asphalt Roofing Industry Bureau

Crowe, Mr. Thomas A.
Greenfeld, Mr. Sidney H.

Bethlehem Steel Corporation

Swanson, Mr. Kenneth R.

Calcium Chloride Institute

Tomes, Mr. Lewis A.

Consumers Union of U.S., Inc.

Kanagy, Dr. Joseph R.

Control Data Corporation

Buckland, Mr. Stanley F., Jr.

Corn Refiners Association, Inc.

Thomas, Mr. James H.
Vomhof, Dr. Daniel W.

Corning Glass Works

Justice, Mr. Benjamin

Dow Chemical Company

Grenley, Mr. Dallas G.
Hamilton, Mr. Robert M.
O'Dell, Mr. William W., Jr.

Eastman Kodak Company

Prsybylowicz, Dr. Edwin P.

Electrical Testing Laboratories, Inc.

Mohan, Mr. Kshitij

Factory Mutual Research Corporation

de Ris, Dr. John N.
Kappraff, Mr. Ronald M.
Orloff, Mr. Lawrence
Rockett, Dr. John A.
Torrance, Dr. Kenneth E.

International Business Machines Corporation

Cleveland, Mr. Norman G.
Shelton, Mr. Carl A. F.

Japan Electronic Industry Development Association

Yamadori, Mr. Yuji

Manufacturing Chemists Association

Brucksch, Mr. William F., Jr.
Clark, Mr. Joseph E.
Herndon, Mr. John L., III

Martin Marietta Corporation (Research Institute for Advanced Studies)

Mengelberg, Dr. Hans-Dieter

Ministere des Postes et Telecommunications (France)

Anquetil, Mr. Phillippe L.

Nippon Electric Company (Japan)

Inawashiro, Mr. Tutomu

Owens-Corning Fiberglas Corporation

Fitch, Mr. William E.

Porcelain Enamel Institute

Burdick, Mr. Milton D.
Gugeler, Mr. Lauren A.
Rushmer, Miss Margaret A.

Structural Clay Products Institute

Johnson, Mr. Paul V.
Watstein, Mr. David

Underwriters Laboratories, Inc.

Castino, Mr. Guy T.

U.S. Navy Marine Engineering Laboratory

Arora, Dr. Om P.

U.S. Steel Corporation

Martin, Mr. John F.

Foreign Guest Workers and Their Sponsors During Fiscal Year 1968

<i>Name</i>	<i>Country</i>	<i>Sponsor</i>
Martins, J. Walters-----	Brazil-----	CAPES (Brazilian NSF).
Cohen, Agda Artna-----	Canada-----	Nuclear Data Project, Oak Ridge, Tenn.
Herman, J. A-----	Canada-----	National Research Council, Canada.
Herman, K. M-----	Canada-----	National Research Council, Canada.
Goonetilleke, H. L-----	Ceylon-----	Dept. of Commerce, Colombo, Ceylon.
Quinn, Terrence J-----	England-----	National Physical Laboratory.
Artru, Madame M. C-----	France-----	Observatoire de Paris, Meudon.
Hanauer, Jean-Pierre-----	France-----	Delegation Generale a la Recherche Scientifique et Technique, Paris.
Guetrot, Alain-----	France-----	Delegation Generale a la Recherche Scientifique et Technique, Paris.
Haddad, Roger J-----	France-----	Delegation Generale a la Recherche Scientifique et Technique, Paris.
Boisrobert, C. Y-----	France-----	Delegation Generale a la Recherche Scientifique et Technique, Paris.
Straub, Johann-----	Germany-----	Institut fur Technische Thermodynamik.
Husmann, E-----	Germany-----	NATO Fellow.
Dajani, Khawla-----	Iran-----	American University.
Goldschmidt, Zvi-----	Israel-----	None.
Natali, Sergio-----	Italy-----	None, employee of Institut of Physics, Univ. of Bari, Italy.
Inamatsu, Teruko-----	Japan-----	National Research Laboratory of Metrology, Japan.
Kaarl, R-----	Netherlands-----	Dutch Service of Weights and Measures.
Olaussen, Leif K-----	Norway-----	Norwegian Government.
Adil, Syed M. F-----	Pakistan-----	United Nations Fellow.
Angeles, Rosalie-----	Philippines-----	American University.
Czyz, Wieslaw-----	Poland-----	American University NSF Grant.
Youssef, Samir-----	UAR-----	International Atomic Energy Agency.
Radovanovic, Dusan-----	Yugoslavia-----	National Academy of Sciences.

ADVISORY COMMITTEES

STATUTORY VISITING COMMITTEE

The Statutory Visiting Committee was established by Act of Congress to advise the Secretary of Commerce and the Director of NBS. This committee is appointed by the Secretary of Commerce. It meets at the call of its chairman. Dates indicate expiration of appointment.

Dr. Robert L. Sproull, Vice President Academic Affairs, Cornell University (1971), Chairman

Dr. Paul C. Cross, President, Mellon Institute (1969)

Dr. Elmer W. Engstrom, President, Radio Corporation of America (1968)
Prof. Norman F. Ramsey, Department of Physics, Harvard University (1970)
Dr. E. R. Piore, Vice President, Research and Engineering, International Business Machines Corporation (1972)

TECHNICAL ADVISORY PANELS

The panels advisory to the National Bureau of Standards were established in 1959 under the terms of a contract between the National Bureau of Standards and the National Academy of Sciences. The Advisory Panels are responsible to the National Academy of Sciences-National Academy of Engineering-National Research Council, Division of Physical Sciences. One panel is established for each of the three Institutes of the Bureau, and one panel for each of the major divisions within the Institutes. The advisory panels to the Institute for Applied Technology are appointed by the President of the National Academy of Engineering. All other panels are appointed by the President of the National Academy of Sciences. The divisional panels ordinarily have eight members although by special arrangement a few have more or less. Terms are usually three years.

Institute for Basic Standards

Advisory Panel to Institute for Basic Standards

Prof. John Todd, California Institute of Technology
Dr. William G. Amey, Leeds & Northrup Company
Dr. J. H. Webb, Eastman Kodak Company
Prof. S. R. Beitler, American Society of Mechanical Engineering
Prof. John Ross, Massachusetts Institute of Technology
Dr. Wade L. Fite, University of Pittsburgh
Dr. Norman D. Coggeshall, Gulf Research & Development Company
Dr. Bruce H. Billings, Aerospace Corporation
Dr. Peter T. Demos, Massachusetts Institute of Technology
Dr. Paul D. Coleman, University of Illinois

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Prof. Philip J. Davis, Brown University
Prof. Charles R. DePrima, California Institute of Technology
Dr. Ralph E. Gomory, International Business Machines Corp.
Prof. M. H. Martin, University of Maryland
Dr. J. Barkley Rosser, University of Wisconsin
Prof. John W. Tukey, Princeton University

Advisory Panel to Electricity Division

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Prof. Raymond M. Fuoss, Yale University
Prof. H. N. Hayward, University of Illinois
Mr. William J. Johnson, Philadelphia Electric Company
Prof. George B. Hoadley, North Carolina State University
Prof. Clyde A. Hutchison, Jr., The University of Chicago
Dean R. B. Lindsay, Brown University
Mr. Douglas C. Strain, Electro Scientific Industries, Inc.

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Mr. B. R. Buus, General Electric Company
Dr. Alsoph H. Corwin, The Johns Hopkins University
Mr. J. K. Emery, The Van Keuren Company
Dr. Robert E. Hopkins, University of Rochester
Mr. Louis Polk, Dayton, Ohio
Mr. Eric J. Schneider, Engis Equipment Company
Dr. George J. Zissis, University of Michigan

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Dr. B. B. Dayton, Consolidated Vacuum Corporation
Prof. Cyril M. Harris, The Columbia University
Prof. Arthur T. Ippen, Massachusetts Institute of Technology
Dr. Harry F. Olson, Radio Corporation of America
Prof. R. S. Rivlin, Lehigh University
Dr. M. E. Shank, Pratt & Whitney Aircraft

Advisory Panel to Heat Division

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Prof. G. B. Benedek, Massachusetts Institute of Technology
Prof. Howard W. Emmons, Harvard University
Dr. E. F. Hammel, University of California
Dr. John P. McCullough, Mobil Oil Corporation
Dr. James W. Moyer, Northrop Corporation
Prof. John G. Phillips, University of California
Dr. Howard Reiss, North American Aviation Science Center

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Prof. Kurt Dressler, Princeton University
Dr. Leo Goldberg, Harvard College Observatory
Prof. Richard C. Lord, Massachusetts Institute of Technology
Prof. Robert Novick, Columbia Radiation Laboratory
Prof. W. E. Spicer, Stanford University

Advisory Panel to Radio Standards Engineering Division

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Dr. George Birnbaum, North American Aviation Science Center
Prof. Sanborn C. Brown, Massachusetts Institute of Technology
Dr. C. Lester Hogan, Motorola, Inc.
Dr. A. O. McCoubrey, Varian Associates
Mr. Frank McGinnis, Sperry Gyroscope Company
Dr. J. A. Pierce, Harvard University
Dr. Bernard M. Oliver, Hewlett-Packard Company
Mr. Theodore S. Saad, Sage Laboratories, Inc.
Mr. D. F. Schmit, Radio Corporation of America
Prof. J. H. Van Vleck, Harvard University
Mr. C. E. White, AVCO Corporation

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Dr. James E. Drummond, Boeing Scientific Research Laboratories
Dr. John W. Evans, Air Force Cambridge Research Laboratory
Dr. Wade L. Fite, University of Pittsburgh
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Dr. John A. Hornbeck, Sandia Corporation
Prof. Robert Novick, Columbia University

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Dr. Arthur Bueche, General Electric Research Laboratory
Dr. N. B. Hannay, Bell Telephone Laboratories, Inc.
Dr. Raymond F. Boyer, Dow Chemical Company
Prof. Roman Smoluchowski, Princeton University
Dr. J. H. Crawford, Chairman, University of North Carolina

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Mr. M. D. Cooper, General Motors Corporation
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Dr. John F. Flagg, American Cyanamid Company
Prof. George Morrison, Cornell University
Prof. L. B. Rogers, Purdue University
Dr. Ralph E. Thiers, Duke University Medical Center

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Dr. William P. Slichter, Bell Telephone Laboratories
Prof. Walter Stockmayer, Dartmouth College
Dr. Howard W. Starkweather, Jr., E. I. Du Pont de Nemours & Co.
Dr. Duane F. Taylor, University of North Carolina
Prof. Bruno H. Zimm, University of California

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Dr. Julius J. Harwood, Ford Motor Company
Dr. Richard A. Oriani, United States Steel Corp.
Dr. Robb M. Thomson, Advanced Research Projects Agency
Prof. David Turnbull, Harvard University
Dr. H. G. F. Wilsdorf, University of Virginia

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Dr. Joseph E. Burke, General Electric Company
Dr. Harris M. Burte, Wright-Patterson Air Force Base
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Dr. J. S. Kasper, General Electric Company
Prof. John L. Margrave, Rice University
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Dr. F. G. Ciapetta, W. R. Grace and Company
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Prof. Robert Gomer, The University of Chicago
Prof. B. S. Rabinovitch, University of Washington
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Institute for Applied Technology

Advisory Panel to Institute for Applied Technology

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Dr. Robert A. Hechtman, McLean, Virginia
Dr. Leon Podolsky, Pittsfield, Massachusetts
Prof. Philip Morse, Massachusetts Institute of Technology
Dr. William W. Eaton, Washington, D.C.
Dr. W. J. Harris, Jr., Battelle Memorial Institute
Dr. Carl H. Madden, U.S. Chamber of Commerce
Dean Joseph R. Passonneau, Skidmore, Owens & Merrith
Mr. Jacob Rabinow, Bethesda, Maryland
Mr. Paul Strassman, National Dairy Products Corp.
Dr. Michael Witunski, St. Louis, Missouri

Advisory Panel to Building Research Division

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Mr. Edward N. Davis, Underwriters' Laboratories, Inc.
Dr. J. Vincent Fitzgerald, Tile Council of America, Inc.
Prof. Hoyt C. Hottel, Massachusetts Institute of Technology
Mr. William H. Lindsay, Jr., Department of Licenses & Inspections, Philadelphia
Mr. Joseph H. Newman, Tishman Research Corporation
Mr. Alwin B. Newton, Borg-Warner Corporation
Mr. Raymond C. Reese, Toledo, Ohio
Dean Charles E. Schaffner, Polytechnic Institute of Brooklyn
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Mr. Herbert H. Swinburne, Nolan, Swinburne and Associates
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Mr. T. E. Werkema, The Dow Chemical Company

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Mr. J. A. Caffiaux, Electronic Industries Association
Mr. Ralph E. Clarridge, IBM Corporation
Mr. Ivan G. Easton, General Radio Company
Mr. Edward S. Hill, Metals and Controls, Inc.
Dr. C. H. Hoffman, Illinois Institute of Technology
Dr. Robert Jeffries, Data Control Systems, Inc.
Mr. H. J. Luer, Bell Telephone Laboratories
Dr. Russell H. Lyddane, General Electric Company
Mr. John S. Norton, Honeywell, Inc.
Mr. Peter R. Perino, Statham Instruments, Inc.
Dr. Robert Pritchard, Stanford Electronics Laboratories
Mr. Robert I. Scace, General Electric Company
Mr. Samuel H. Watson, Radio Corporation of America
Dr. Richard C. Webb, Colorado Instruments, Inc.

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Prof. Russell L. Ackoff, University of Pennsylvania
Prof. George B. Dantzig, Stanford University
Mr. Leslie C. Edie, Port of New York Authority
Mr. Martin L. Ernst, Arthur D. Little, Inc.
Dr. Hugh J. Miser, The Travelers Research Center, Inc.
Mr. David Novick, The Rand Corporation
Prof. Thornton L. Page, Wesleyan University
Dr. George Pettee, Research Analysis Corporation
Prof. Gustave J. Rath, Northwestern University
Prof. R. F. Rinehart, U.S. Naval Postgraduate School

Advisory Panel to Center for Computer Sciences and Technology

Dr. William W. Eaton, Washington, D.C., Chairman
Mr. John Diebold, The Diebold Group, Inc.
Mr. Robert B. Forest, DATAMATION Magazine
Prof. Walter F. Frese, Harvard University
Dr. Cuthbert C. Hurd, Computer Usage Company, Inc.
Mr. James D. Gallagher, McCall Corporation
Dr. Adrian McDonough, University of Pennsylvania
Dr. Jack Moshman, EBS Management Consultants, Inc.
Dr. Charles A. Phillips, Business Equipment Manufacturers Association
Prof. Alan J. Rowe, University of Southern California
Prof. John Tukey, Princeton University
Dr. Willis H. Ware, The Rand Corporation

Center for Radiation Research

Advisory Panel to Center for Radiation Research

Dr. Peter T. Demos, Massachusetts Institute of Technology, Chairman
Dr. John S. Blair, University of Washington
Mr. Casimer J. Borkowski, Oak Ridge National Laboratory
Dr. Marshall R. Cleland, Radiation Dynamics, Inc.
Dr. Charles J. Mullin, University of Notre Dame
Dr. George F. Pieper, National Aeronautics and Space Administration
Dr. W. C. Roesch, Pacific Northwest Laboratory
Prof. Erwin F. Shrader, Case Institute of Technology

AWARDS AND HONORS

Recognition of the Bureau's contributions to science and technology often takes the form of awards and honors from Government, academic, professional, and industrial groups. The following list reflects such recognition bestowed on Bureau staff members during fiscal year 1968¹:

<i>Recipient</i>	<i>Award</i>
Alexander, S. N.	Harry Good Memorial Award, American Federation of Information Processing Societies
Astin, A. V.	Commendation Plaque, National Conference on Weights and Measures Distinguished Alumni Award, University of Utah Alumni Association
Brenner, F. Cecil	Notable Services Award, Apparel Research Foundation
Cutkosky, Robert D.	Scientific Achievement Award, Washington Academy of Sciences
Harris, Forrest K.	Edward Bennett Rosa Award
Hoffman, John D.	Samuel Wesley Stratton Award
Hougen, Jon T.	Coblentz Society Award
Jensen, Malcolm W.	Commendation Plaque, National Conference on Weights and Measures Hands of Cooperation Award, National Forest Products Association
Marsden, Charles P., Jr.	Award of Merit, American Society for Testing and Materials
Meinke, W. Wayne	American Nuclear Society 1968 Special Award for Industrial Applications for Radiation Techniques
Meyerson, Melvin R.	George Kimball Burgess Award, Washington Chapter of the American Society for Metals
Stiehler, Robert D.	Award of Merit, American Society for Testing and Materials
Williams, Morgan L.	Bissell Award, Washington Section of the American Welding Society

EDUCATION, TRAINING, AND UNIVERSITY LIAISON

A broad employee development program, ranging from broad surveys to very detailed treatment of a new or specialized area of research, is available to all staff members. Courses and seminars are implemented primarily through the NBS Graduate School and non-Government educational and training facilities. The programs cover education through postdoctoral research, and are offered at both the Boulder and Gaithersburg Laboratories. The primary objectives are to increase employee knowledge, skills and efficiency in assigned duties and to prepare Bureau staff members to respond to the rapidly changing technology at all levels. Programs also include emphasis on maintaining liaison with the public, industry, commerce and science.

¹ Commerce Department Medal Awards, previously distributed in February of each year, will now be distributed in October. The 1968 listing of award recipients will appear in the 1969 Technical Highlights.

In addition, the Bureau is becoming more and more involved with projects in the community interest. The Hood College Program for Women at the National Bureau of Standards, supported by the National Science Foundation, has made expert instruction and facilities available to five participating small liberal arts colleges. A course in science of metals for high school teachers using Bureau expertise and facilities, in cooperation with the American Society for Metals was given in the Fall Semester. In addition to numerous seminars offered at the division and institute level, there is a weekly Scientific Staff Meeting at which current topics of broad interest to the Bureau are presented. These are open to the public and are attended by members of the universities, industry and Government community, as well as by NBS personnel. Speakers are drawn from the outside community as well as from within the Bureau. Monthly colloquia under the joint sponsorship of the National Institutes of Health and the National Bureau of Standards are also offered, with each institution alternating as host.

NBS Graduate School

The NBS Graduate School curriculum includes graduate and undergraduate courses in the physical sciences, mathematics and specialized branches of engineering. A series of scientific colloquia and seminars designed to update and continue the education of the postdoctoral scientist are led by research leaders from the Bureau and from other research centers and universities. In addition, general staff development courses are offered, such as Scientific Russian, Supervision and Management, Reading Improvement, Technical Report Writing, and administrative and clerical conferences and workshops.

Two special programs, designed for technicians and subprofessional laboratory personnel, offer courses both in-house and in cooperation with the Montgomery Junior College leading to two NBS Technician Certificates and/or the A. A. degree at the Montgomery Junior College. Surveys periodically redetermine course offerings and keep the program in step with the changes and variations in educational requirements and the changing technology. Graduate degrees based partly on credit obtained for courses or thesis research carried on under the NBS Graduate School Program have been earned by 4 Bureau employees this year, bringing the total to 346 graduate degrees earned at 45 different universities since the establishment of the educational program in 1908.

The Graduate School at Boulder is associated with the University of Colorado in a Joint-Course program and Adjunct Professor Plan. Various graduate departments of the NBS Graduate School at Boulder and the University offer courses which mutually benefit the Government and the University.

Postdoctoral Research Associateships

The National Bureau of Standards, in cooperation with the National Research Council, National Academy of Sciences-National Academy of Engineering, offers a number of awards for postdoctoral research.

These awards provide young scientists of unusual ability and promise an opportunity for fundamental research in various branches of the physical, engineering, and mathematical sciences. Applications are evaluated by a Board of Selection appointed by the National Research Council. The NRC-NBS Postdoctoral Research Associateship program has been in existence since 1954. There have been 173 awards made during these years; currently there are 26 in residence.

Postdoctoral Research Associates on Duty During Fiscal Year 1968

NAME	UNIVERSITY	NBS ADVISER
Anderson, Robert	U. of Calif. (Berkeley)	T. Carrington
Balling, Ludwig	Harvard	R. C. Mockler
Cahill, Kevin	Harvard	S. Meshkov
Cooper, Martin	Brandeis University	M. S. Green
Davis, Douglas	U. of Florida	H. K. Okabe
DeLancey, George	U. of Pittsburgh	H. Oser
Duerst, Richard	U. of Calif. (Berkeley)	G. F. Kokoszka
Ensign, Thomas	U. of Wyoming	T. Chang
Fickett, Frederick	Oregon State University	R. Powell
Fong, Jeffrey	Stanford University	H. Oser
Golub, Stephen	Columbia University	B. Steiner
Gould, Harvey	U. of Calif. (Berkeley)	K. Shuler
Greenhouse, Jeffrey	U. of Calif. (Berkeley)	W. J. Lafferty
Hadley, Steven	U. of Calif. (Davis)	R. Keller
Hartl, Werner	Columbia University	R. Howard
Hettche, Leroy	Carnegie	L. Irwin
Hoegy, Walter	U. of Michigan	M. S. Green
Kelly, Robert L.	U. of Calif. (Berkeley)	C. Muehlhause
Latanision, Ronald	Ohio State University	A. W. Ruff
McAlister, Archie	U. of Maryland	J. Cuthill
Miller, Kenneth	Iowa State University	M. Krauss
Manson, Steven	Columbia University	A. W. Weiss
Negas, Taki	Ohio State University	R. S. Roth
Osgood, Charles	U. of Calif. (Berkeley)	M. Newman
Parke, William	George Washington University	R. W. Hayward
Pechukas, Philip	U. of Chicago	K. Shuler
Plummer, Earl	Cornell University	R. D. Young
Pong, William	U. of Cincinnati	R. P. Madden
Ponzini, Robert	Michigan State University	S. Meshkov
Pringle, Wallace	MIT	W. J. Lafferty
Radebaugh, Ray	Purdue University	D. Chelton
Raveche, Harold	U. of California (LaJolla)	K. Shuler
Retajczyk, Theodore	MIT	J. Taylor
Robertson, Baldwin	Washington University	R. Rubin
Shirk, James	U. of Calif. (Berkeley)	A. M. Bass
Stemmler, Rosemarie	U. of Illinois	M. Newman
Sullivan, Donald	Vanderbilt University	R. A. Kamper
Sullivan, Peter	Rensselaer Polytechnic Inst.	J. D. Hoffman
Veillon, Claude	U. of Florida	M. Margoshes
Weisman, Irwin	U. of California (Berkeley)	L. H. Bennett
Williams, Harry	U. of Virginia	M. Danos
Zalewski, Edward	U. of Chicago	R. A. Keller

Non-Government Education

Non-Government education falls into three categories . . . full-time (3 to 12 months) graduate study and research assignments at universities and research centers; full-time (less than 3 months) attendance at institutes, seminars, short concentrated courses and workshops; and part-time, job related academic courses at universities and in industry. In the last year 452 staff members at Washington and Boulder were trained through non-Government facilities, and

13 career employees were selected for full-time graduate study or research assignments at universities and research centers. Participants in approved full-time training programs receive full salary and expenses, including tuition, fees, travel, and per diem, as well as transportation of family and household effects. In addition, 434 staff members, mostly from technical divisions, attended job-related courses on a semester basis, and shorter concentrated courses at universities and in industry.

Interagency Training

Courses made available through the Interagency Training Programs are an additional effective means of improving program operations for NBS personnel. Courses are offered at Government facilities in Supervision, Management, Office Skills and Practices, and specialized programs. This pooling of agency resources offers broader employee development opportunities at a saving to the Government. During 1968, 212 National Bureau of Standards employees took advantage of the interagency offerings.

PUBLICATIONS*

PUBLICATIONS IN THE BUREAU'S SERIES

During the year NBS publications totaled 998 published papers and documents.

Of the formal publications, 111 appeared in the *Journal of Research*, and 546 in the journals of professional and scientific societies. Also, 249 summary articles were presented in the Bureau's monthly *Technical News Bulletin*.

In the nonperiodical series, 86 documents were published: 10 in the Monograph series, 3 in the Handbook series, 11 in the Miscellaneous Publication series, 3 in the Special Publication series, 44 in the Technical Note series, 11 in the National Standard Reference Data Series, and 7 in the Building Science Series.

Journal of Research. Contains full research papers, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Advances in measurement standards and techniques . . . physical constants . . . properties of materials . . . instrumentation . . . radio propagation.

The *Journal* is published in three separate sections . . .

- A. Physics and Chemistry, issued six times a year. Annual subscription: Domestic, \$6; foreign, \$7.25; single copy, \$1.00.
- B. Mathematical Sciences (Name changed from Mathematics and Mathematical Physics to Mathematical Sciences effective January 1968), issued quarterly. Annual Subscription: Domestic, \$2.25; foreign, \$2.75; single copy, 75 cents.
- C. Engineering and Instrumentation, issued quarterly. Annual subscription: Domestic, \$2.75; foreign, \$3.50; single copy, 75 cents.

*Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (foreign postage, one-fourth additional). The NBS non-periodical series are also available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151. Reprints from outside journals and the NBS *Journal of Research* may often be obtained directly from the authors.

Volume 71A (Phys. and Chem.), No. 4 (July–Aug. 1967)

- Kinetics of crystallization in multicomponent systems: I. Binary mixtures of *n*-paraffins. John I. Lauritzen, Jr., Elio Passaglia, and E. A. DiMarzio.
- Kinetics of crystallization in multicomponent systems: II. Chain-folded polymer crystals. John I. Lauritzen, Jr., and Elio Passaglia.
- Infrared absorption spectra of 2-oxo-1, 3-bis(phenylhydrazono) derivatives and related bis- and tris-phenylhydrazones. Alexander J. Fatiadi.
- Dielectric constant of *n*-hexane as a function of temperature, pressure, and density. Frederick I. Mopsik.
- Heat capacities and related thermal data for diethyl phthalate crystal, glass, and liquid to 360 °K. S. S. Chang, J. A. Horman, and A. B. Bestul.
- Heats of formation of aluminum diboride and α -aluminum dodecaboride. Eugene S. Domalski and George T. Armstrong.
- Effect of environment upon the melting point of Al_2O_3 . S. J. Schneider and C. L. McDaniel.

Volume 71A (Phys. and Chem.), No. 5 (Sept.–Oct. 1967)

- Zeeman effect in positronium annihilation at low temperatures. William C. Saunder and Richard D. Deslattes.
- Surface-layer relaxation in the dielectric spectrum of CaF_2 doped with GdF_3 . A. D. Franklin, S. Marzullo, and J. B. Wachtman, Jr.
- Thermal expansion of rutile from 100 to 700 °K. Richard K. Kirby.
- Mass spectrometric study of photoionization. VII. Nitrogen dioxide and nitrous oxide. Vernon H. Dibeler, James A. Walker, and Susan K. Liston.
- Ionization constants of substituted benzoic acids in ethanol-water. G. M. Brauer, George Durany, and Harold Argentar.
- Dissociation constants of some substituted nitrophenols in aqueous solution at 25 °C. R. A. Robinson.

Volume 71A (Phys. and Chem.), No. 6 (Nov.–Dec. 1967)

- William Frederick Meggers, A Tribute. Karl G. Kessler.
- The second spectrum of ytterbium (Yb II). W. F. Meggers. (Edited by Charlotte E. Moore.)
- An improved description of technetium spectra (Tc I and Tc II), 2000 to 9000 Å. W. R. Bozman, W. F. Meggers, and C. H. Corliss.
- Lifetimes of energy levels in neutral iron. C. H. Corliss and J. L. Tech.
- Transition probabilities in argon I. C. H. Corliss and J. B. Shumaker, Jr.
- The fifth spectrum of praseodymium. Victor Kaufman and Jack Sugar.
- Fundamental energy levels of neutral promethium (Pm I). Joseph Reader and Sumner P. Davis.

Volume 72A (Phys. and Chem.), No. 1 (Jan.–Feb. 1968)

- Reactions of uranium with the platinide elements. I. The uranium-ruthenium system. John J. Park.
- Reactions of uranium and the platinide elements. II. The uranium-rhodium system. John J. Park.
- Reactions of uranium and the platinide elements. III. The uranium-iridium system. John J. Park and Lyle R. Mullen.
- Phase relations between palladium oxide and the rare earth sesquioxides in air. C. L. McDaniel and S. J. Schneider.
- Preparation and purification of some oxidation products of perylene. Alexander J. Fatiadi.

Electron spin resonance spectra of polymer radicals in aqueous solution. Roland E. Florin, Fred Sicilio, and Leo A. Wall.

A mass spectrometric study of the BeO-BeF₂ system at high temperatures. J. Efimenko.

Laser induced Raman spectra of some tungstates and molybdates. R. K. Khanna, W. S. Brower, B. R. Guscott, and E. R. Lippincott.

Valence—only correlation in LiH and BeH⁺. W. A. Sanders and M. Krauss.

Thermalization by elastic collisions: positronium in a rare gas moderator. William C. Sauder.

Density fluctuations in fluids having an internal degree of freedom. Raymond D. Mountain.

Volume 72A (Phys. and Chem.), No. 2 (Mar.-Apr. 1968)

Constant pressure flame calorimetry with fluorine II. The heat of formation of oxygen difluoride. Reatha C. King and George T. Armstrong.

The heat of formation of boron carbide. E. S. Domalski and G. T. Armstrong.

Solubility of tris(hydroxymethyl)aminomethane in water-methanol solvent mixtures and medium effects in the dissociation of the protonated base. Paul Schindler, R. A. Robinson, and Roger G. Bates.

Calibration of the nickel dimethylglyoxime spectral shift at pressures to 20 kilobars for use in spectroscopic pressure measurement. Helen W. Davies.

Second virial coefficient of He⁴ in the temperature range from 2 to 20 °K. Marjorie E. Boyd, Sigurd Y. Larsen, and Harmon Plumb.

Diffusion rates in inorganic nuclear materials. A. L. Dragoo.

Effect of oxide additions on the polymorphism of tantalum pentoxide. (system Ta₂O₅-TiO₂). J. L. Waring and R. S. Roth.

Effect of statistical counting errors on wavelength criteria for x-ray spectra. John S. Thomsen and F. Y. Yap.

Thermodynamic properties of ammonia as an ideal gas. Iester Haar.

Volume 72A (Phys. and Chem.), No. 3 (May-June 1968)

Jump rates for point defects in special positions held by a trapping center of noncubic symmetry. H. S. Peiser and J. B. Wachtman, Jr.

The thermodynamics of the ternary system: water-potassium chloride-calcium chloride at 25 °C. R. A. Robinson and A. K. Covington.

Force field for SiF₄. Ira W. Levin and Stanley Abramowitz.

Blemish formation in processed microfilm. C. I. Pope.

Absolute isotopic abundance ratios of common, equal-atom, and radiogenic lead isotopic standards. E. J. Catanzaro, T. J. Murphy, W. R. Shields, and E. L. Garner.

Role of nickel in Al-10 percent Si composites containing nickel-coated sapphire whiskers. H. Yakowitz, W. D. Jenkins, and H. Hahn.

Volume 71B (Math. and Math. Phys.), No. 2 and 3 (Apr.-Sept. 1967)

Poincare's conjecture is amplified by a conjecture on free groups. Roger D. Traub.

Construction of *EPr* generalized inverses by inversion of nonsingular matrices. John Z. Hearon.

A generalized matrix version of Rennie's inequality. John Z. Hearon.

Polar factorization of a matrix. John Z. Hearon.

Two classical theorems on commuting matrices. Morris Newman.

A converse to Banach's contraction theorem. Philip R. Meyers.

E-transforms (II). F. M. Ragab.

Criterion for the stability of numerical integration methods for the solution of systems of differential equations. Abbas I. Abdel Karim.
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PATENTS

The following U.S. Patents have been granted to Commerce inventors and assigned to the United States of America, as represented by the Secretary of the Department noted in parentheses:

Hoynes, Dagfin S. No. 3,329,957, July 4, 1967. Antenna System Employing Human Body as Radiator (Commerce).

- Armstrong, Richard W. No. 3,331,228, July 18, 1967. Combination Lock (GSA).
- Bruck, Stephen D. No. 3,331,656, July 18, 1967. Chemically Crimping Nylon Fibers Through Formation of Disulfide Bonds Therein (Commerce).
- Weitzel, Daniel H. No. 3,339,948, September 5, 1967. Pipe Coupling (Air Force).
- Selby, Myron C. No. 3,354,411, November 21, 1967. Coaxial Transmission Line T-Junction Having Rectangular Passageway Dimensioned Beyond Cutoff for Higher Order Modes (Commerce).
- Jones, Frank E. and Castle, Alfred B. Sr. No. 3,354,544, November 28, 1967. Method and Apparatus for Making Electrical Elements (Navy).
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- Boynton, Ira D. and Bode, Robert H. No. 3,378,257, April 16, 1968. Apparatus for Moving and Orienting Limp Pieces of Materials (Commerce).

